ASTM Bulletin

NEWS—ASTM Corrosion Program; Actions on Standards; Publications; Award of Merit; Case Histories; Student Prize Awards and Junior Members.

PAPERS—Wood; Cement and Concrete; Powder Metallurgy Alloys; Fatigue; Specular Gloss; Adhesives; Adhesive Bonds; Viscosity Measurements.

American Society for Testing Materials

ASTM BULLETIN

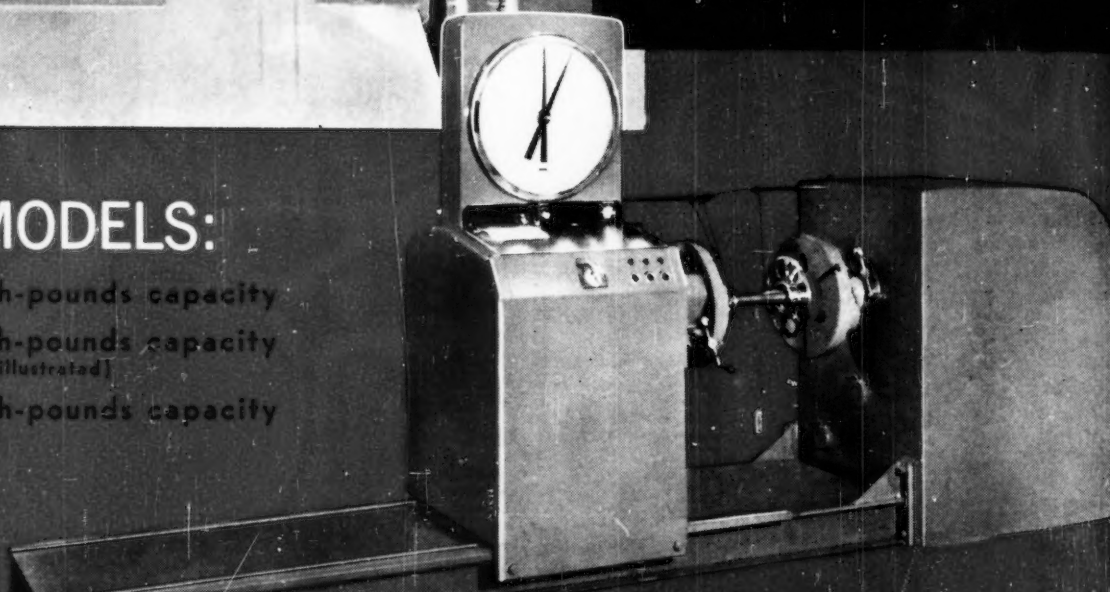*Published by*
AMERICAN SOCIETY for TESTING MATERIALS

This Issue Contains

ASTM Bulletin is indexed regularly by Engineering Index, Inc.

Telephone: Rittenhouse 6-5315   Cable Address: Testing, Philadelphia

OCTOBER—1950
No. 169

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ASTM BULLETIN

"Promotion of Knowledge of Materials of Engineering, and Standardization of Specifications and Methods of Testing"

ASTM Exposure Test Site Program

A BILLION dollars is a conservative estimate of the loss each year due to the corrosion of metals. The effects of atmospheric deterioration on plastics, rubber, textiles, wood, and other engineering materials would more than double this figure.

The American Society for Testing Materials has long recognized the tremendous economic significance of these problems, and has been one of the foremost contributors to knowledge in these fields by conducting authoritative and impartial atmospheric and other exposure tests that have provided engineers with reliable data for design and operation of structures and equipment of all kinds. This article, after briefly reviewing the Society's accomplishments in a period of some forty years, presents plans for placing ASTM Exposure Testing on a firm, long-time basis, to assure a continued flow of authentic data on deterioration characteristics of a wide variety of materials, ranging from the various ferrous and non-ferrous metals and their alloys through paints, ceramics, rubber, adhesives, plastics, textiles, wood, and "composite" constructions, and invites the financial as well as technical support of the membership and of industry.

Atmospheric Exposure Tests

The Society's work on atmospheric exposure tests dates back more than 40 years to the formation of the committees now designated as Committee D-1 on Paint, Varnish, Lacquer, and Related Products and Committee A-5 on Corrosion of Iron and Steel. In 1906 the first-named committee started the now classic tests of paints on the Pennsylvania Railroad Bridge at Havre de Grace, and shortly thereafter the second committee began exposure tests at Pittsburgh of twelve samples of galvanized wire with different weights of coatings. Since these early be-

ginnings the Society, through the activities of seven of its committees, now has more than 25,000 test specimens on exposure at 18 outdoor test sites and expects the number of specimens to double within the next few years. A brief account of this work follows, including reference to some major contributions to engineering knowledge that have resulted[1] and mention of plans for future work.

Committee A-5, organized in 1906 to study the corrosion of iron and steel, has the longest record of exposure testing in the Society. In addition to the early galvanized wire tests above mentioned, it cooperated with Committee D-1 on tests of painted steel fence panels in Atlantic City about 1910, and has also done much work on immersion tests in various types of water. It established its first atmospheric exposure racks on three test sites acquired in 1916, for the purpose of determining effect of steel composition on corrosion resistance, and it was on the strength of these tests that the committee over twenty years ago established the copper requirements of copper-bearing steel for effective resistance to atmospheric corrosion. Some of these tests in the mildly corrosive atmosphere of Annapolis are still continuing after 35 years exposure.

The pros and cons of the sulfuric acid test as a gage of atmospheric corrosion resistance of ordinary ferrous metals were settled by the committee in tests conducted around the year 1930 which proved that this test did not possess consistent repeatability nor did it show correlation with service results.

There followed in the late 20's and early 30's extensive exposure testing of:

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[1] For a more complete record of this work and of its many accomplishments, see Report of Advisory Committee on Corrosion, *Proceedings.* Am. Soc. Testing Mats., Vol. 47, p. 211 (1947).

(1) uncoated and galvanized corrugated sheets with various weights of zinc coatings; (2) metallic-coated hardware, structural shapes, tubular goods, etc., coated with eight commercial coatings (Fig. 1); and (3) wire and wire products, such as unfabricated wire, barbed wire, wire strand, farm-field fence and chain-link fence exposed at eleven test sites from the Atlantic to the Pacific (Fig. 2). While the wire tests are designed primarily for evaluation of zinc coatings of varying weights and processes of application on wires of varying size, they also include comparisons with copper-covered wire, lead-coated wire, and uncoated corrosion-resistant steel wire. Figure 3 illustrates the type of data being obtained from these tests. All three of these series of tests are continuing and have yielded invaluable data on correlation of weight of zinc coating and expected service life that have enabled this committee to write a number of authoritative specifications for zinc-coated products.

Committee A-10, organized in 1929, has given considerable attention to the methods of conducting, and the evaluation of, plant corrosion tests of corrosion- and heat-resisting steels. It is now collecting material for an extensive program for a 15- to 20-year atmospheric exposure test of corrosion-resisting steels which will be initiated as soon as the new ASTM test sites are available.

The committee has made periodic inspections of various corrosion-resisting steel installations including trains, deck houses of Navy destroyers, and architectural structures in New York, N. Y., Philadelphia, Pa., and Atlantic City, N. J.

Committee B-3, organized in 1922, has done much on laboratory testing in relation to long-time exposure tests on non-ferrous metals and alloys, partic-

ularly with respect to the salt spray test. In 1931 it initiated a long-time series of atmospheric exposure tests of representative non-ferrous metals and alloys (Fig. 4) at nine widely separated sites throughout the United States. Ten-year results have been reported and the tests are still continuing. Much valuable information has been obtained on the effect of electrolytic couple action under different conditions on the corrosion of ferrous and non-ferrous metals in various combinations (Fig. 4). Galvanic couples of aluminum, copper, mild steel, tin, zinc, nickel, and lead were exposed in 1931 couples of these metals with corrosion resisting steels in and steel couples in 1941. These extensive tests have recently been supplemented by a third series of couples involving the newer aluminum and magnesium alloys.

New work by this committee includes calibration of atmospheres at various locations by weight-loss tests on zinc and steel specimens and—jointly with Committee B-7—atmospheric exposure of various aluminum and magnesium alloys.

Committee B-6 was organized in 1930 from a group that had earlier been working as a subcommittee of Committee B-2. In 1929 it began exposure tests of zinc-base and aluminum-base die-casting alloys that were extensively supplemented in later years and are still under way. Tests of upwards of 35,000 specimens of 21 different alloys have enabled the committee to establish satisfactory specifications for the best of these alloys and to correlate accelerated laboratory test procedures with long-time exposure tests. New
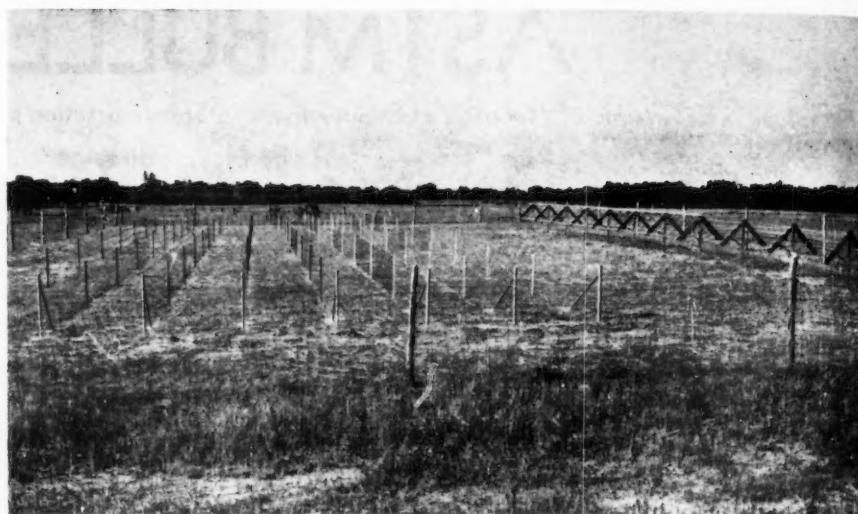
Fig. 2.—Wire and Wire Products at Manhattan, Kansas.

atmospheric exposure tests are being planned.

Committee B-7, organized in 1928, has developed several tests for anodic coatings on aluminum. It has recently, in cooperation with Committee B-3, developed an important program for long-term atmospheric exposure testing of cast and wrought aluminum and magnesium alloys and will use the new test site facilities of the Society.

Committee B-8, organized in 1941, has already extensively studied corrosion resistance of various electro-deposited metallic coatings, including lead coatings on steel and copper-nickel-chromium coatings on high-carbon steel which have enabled the committee to write important specifications for several such coatings.

Plans have been made for extensive exposure tests of five types of chromate treatments for various metallic finishes.

Committee D-1, organized in 1902, established by extensive service tests with paint on steel the now generally accepted principles of primer paint formulation. The record of the Havre de Grace tests previously mentioned is still worthy of study by anyone interested in large-scale service tests. In recent years this committee has exposed panels in various localities to study the effects of special surface treatments of steel on paint service and to correlate the results of accelerated laboratory tests with outdoor exposure. Exposure tests of white paints, anti-fouling paints, and varnishes have been made.

The committee has on exposure a number of wooden specimens (Fig. 5) for evaluating dirt retention properties and is presently considering a program to establish geographic or weather zones of behavior of metal protective paints.

Other Committees now becoming interested in exposure testing to determine deterioration characteristics of materials are Committees D-19 on Industrial Water, C-19 on Structural Sandwich Constructions, D-7 on Wood, D-14 on Adhesives, and D-20 on Plastics. Several are now working on test programs.

Administration

The administration of these programs of exposure tests has posed an increasingly difficult problem. Nearly all of the tests so far, as will be evident from the foregoing presentation, are in the field of metals (that is, corrosion), involving a half-dozen committees who at first were reasonably well able to coordinate their work through interlocking membership and by direct
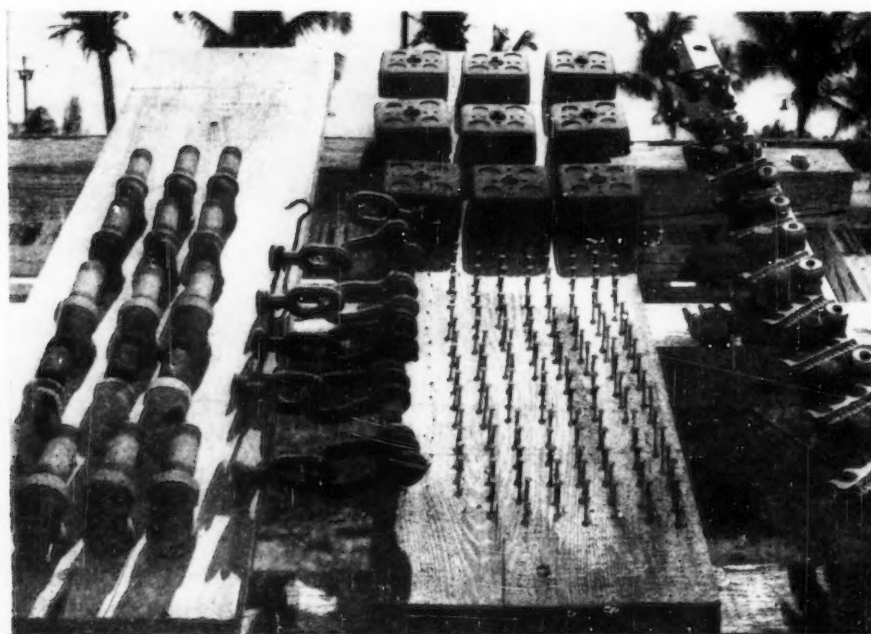
Fig. 1.—Metallic-Coated Hardware Specimens of Committee A-5 at Key West, Fla.

Pittsburgh Groups (——), State College (————), oz. per. sq. ft. of surface.
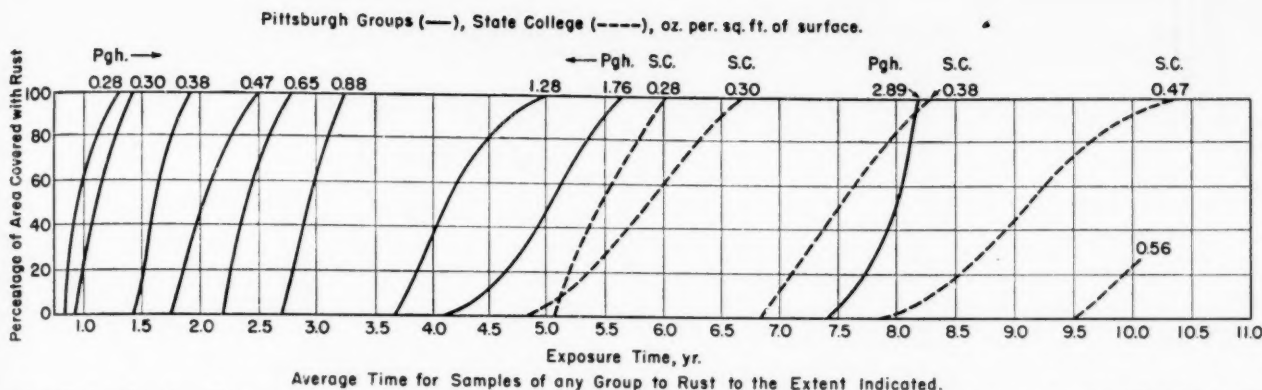
Fig. 3.—Data Showing Progressive Development of Rust on Zinc-Coated Unfabricated Wire of Various Coating-Weight Groups.

cooperation on specific projects of mutual interest. But, as time went on, four of these committees had acquired a total of 22 test sites, most being used jointly, but some exclusively by one committee, having different tenures of occupancy and conditions of use, and overlapping considerably in kind of prevailing atmospheric conditions. Efficient servicing of so many scattered sites was next to impossible through our voluntary committee organization.

About ten years ago it became clear that tighter coordination was essential, and the Society in 1942 organized the Advisory Committee on Corrosion, primarily to coordinate all corrosion researches by metals committees of the Society and to consider simplification of the exposure test site situation. The committee consists of "members-at-large" appointed by the Board of Directors and, initially, two representatives from those technical committees engaged in corrosion studies. Now that committees in the field of nonmetals are seriously planning atmospheric exposure tests, for which test sites under the control of the Society will be well suited, representation on the Advisory Committee is being extended to include other than metals committees.

The Advisory Committee's study of the exposure test site problem was taken up actively at the end of the war. It has now become clear that far too many test sites are being operated and that a much smaller number could be selected that would represent collectively all of the types of atmosphere in which exposure tests are now being made. Instances developed where a test site had become too small with no room for expansion; other sites had to be abandoned because of industrial encroachment (Fig. 6); and still other sites have had to be abandoned and tests interrupted because the space has had to be reclaimed by the owner. Accordingly the Advisory Committee has paid particular attention to long and secure tenure of occupancy, adequate size, and as much

freedom as possible from later encroachment. With the selection of the test sites described below, the Board of Directors has given to the Advisory Committee on Corrosion responsibility for the acquisition and maintenance of ASTM exposure test sites and jurisdiction in all matters having to do with the use of the test sites.

Seven New Test Sites Selected

The Advisory Committee on Corrosion has selected seven test sites representative of various types of atmosphere, large enough to provide for all seeable future needs, and of long tenure of occupancy so far as such can be assured in advance. These are briefly described below and are spotted on the accompanying map, which also shows other test sites that are now in use but will probably be discontinued when tests there are completed. It will be seen that an important addition to the classes of sites already in operation are those in the Canal Zone where both mild and severe tropical atmospheres exist. The possibility also of use, in cooperation with the National Research

Council of Canada, of a site where extreme low temperatures prevail is a most interesting one and completes the wide range of atmospheric conditions available for ASTM exposure testing.

INDUSTRIAL ATMOSPHERE

New York City, N. Y.—Arrangements have been made with the Port of New York Authority for the use of an area of about 20,000 sq. ft. on the roof of the 16-story Authority Building at Eighth Ave. and Fifteenth St. in New York City. The atmosphere is typically industrial and similar to those of Pittsburgh and Altoona where exposure tests have been conducted in the past. Five 57½-ft. pipe frames have already been installed on the Port Authority roof (Fig. 7) and some tests are in progress.

SEMI-INDUSTRIAL ATMOSPHERE

Columbus, Ohio.—Arrangements have been completed with Battelle Memorial Institute for the use of a penthouse section on the roof of a five-story Institute building in Columbus. The area presently available is about 11,000 sq. ft.; other roof areas are available at the Institute if needed later. There are a number of railroad shops approximately two miles south-

Fig. 4.—Galvanic Couples and Tensile Specimens of Non-Ferrous Metals and Alloys

Cooperation with Canada has been arranged through the National Research Council

Ithaca, N.Y.   Bridgeport, Conn.
STATE COLLEGE, PA.   NEW YORK CITY
Sandy Hook, N.J.
Davis, Calif.
POINT REYES, CALIF.
Santa Cruz, Calif.
Ames, Iowa
Pittsburgh, Pa.   Altoona, Pa.
Lafayette, Ind.   COLUMBUS, OHIO   Annapolis, Md.
Manhattan, Kans.
KURE BEACH, N.C.
College Station, Texas
Tela, Honduras
PANAMA CANAL ZONE
PORT ARANSAS, TEXAS
Sites That Had To Be Discontinued
Ft. Sheridan, Ill.   La Jolla, Calif.
Rochester, N.Y.   Phoenix, Ariz.
Pittsburgh (Brunot Island), Pa.
Key West, Fla.

⊚ SITES established under new program
• Other sites now in use

east and the prevailing winds are from this direction. Battelle tests indicate that this is a typical atmosphere intermediate between industrial and rural.

RURAL ATMOSPHERE

State College, Pa.—The long-established test site at Pennsylvania State College, first used by the Society in 1925, furnishes a typical rural atmosphere as borne out by tests extending over the past quarter century. Arrangements have been made to double the area presently being used, giving a total of about 40,000 sq. ft. Many tests are now under way there.

MARINE ATMOSPHERE

Three sites have been chosen, with sea-coast atmospheres representing the major bodies of water surrounding Continental United States.

Atlantic Coast: Kure Beach, N. C.—This site is adjacent to the well-known test facilities operated by The International Nickel Co. at Kure Beach, N. C., and is made available by agreement with that company. At least an acre is available and some tests have been started.

Gulf Coast: Port Aransas, Tex.—Arrangements have been completed with the University of Texas for a site of about an acre on the Gulf of Mexico at Port Aransas, Texas, on land adjoining the laboratories of The Institute of Marine Science of the University. The occurrence of periodic high storm tides in this area will necessitate the use of piling to support our test racks.

Pacific Coast: Point Reyes, Calif.—Ar-

rangements have been completed with American Telephone and Telegraph Co. for the use of approximately one acre of land on their property at Point Reyes, Calif., about 30 miles north of San Francisco. This is an ideal location representative of typical fog conditions of that region.

TROPICAL ATMOSPHERE

Canal Zone.—Arrangements have been completed with the Panama Canal Zone authorities for the use of available space at several locations in the Canal Zone, including Gatun, Miraflores, and Cristobal, which are classed as mildly tropical. Tests are already under way at the Miraflores location.

A severe tropical atmosphere exists on Barro Colorado Island in Gatun Lake, and negotiations are under way with the Smithsonian Institution for suitable space in a section of the island occupied by the Canal Zone Biological Area.

CANADIAN TEST SITES

The Advisory Committee on Corrosion and the National Research Council of Canada have agreed to the reciprocal use of test sites under their respective jurisdictions. The Canadian Council is establishing sites at Halifax (sea coast), Ottawa (rural), Montreal (industrial), and Saskatoon. Consideration is also being given to the establishment of sites on Vancouver Island and at Bakers Lake in the Far North. The A.C.C. has been advised that space could be made available at any of these sites.

The Society's long-range program on exposure tests includes the establishment of several underwater sites. Tentative arrangements have been made for the use of a part of the seawater basin used by The International Nickel Co. at Wrightsville, N. C.

Other sites may be considered at some future time, but it is believed that those now selected will adequately meet the Society's long-time requirements for atmospheric exposure testing.

Financial Support Needed

The Advisory Committee on Corrosion has estimated that the cost of acquiring, maintaining, and supervising the ASTM Exposure Test Sites for the first ten years will be at least $100,000. This includes cost of equipping the sites with all test facilities—test racks, specimen frames, and various special items—enclosing them where necessary, and providing a qualified engineer from Society headquarters staff to give the necessary technical supervision and to service the sites and test specimens under the general guidance of the Advisory Committee and the various technical committees sponsoring the exposure tests. Centralization of this latter service is essential to the efficient operation of the whole program and will
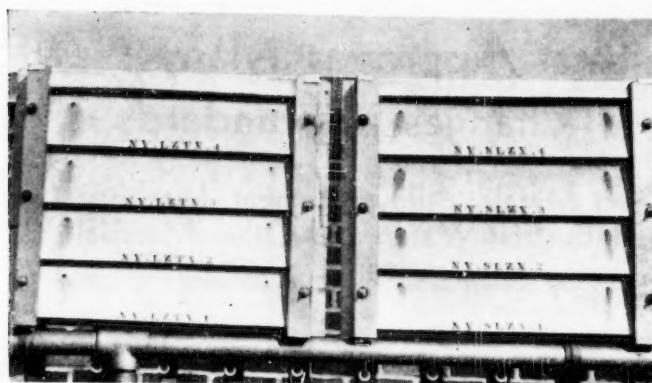
Fig. 5.—Committee D-1 Wooden Panels Exposed for Evaluation of Copper and Iron Stain and Dirt Retention.

relieve committee officers and members of burdensome detail—including the not inconsiderable expense involved—which, with the rapid growth of the program, they could no longer handle effectively. The estimate of $100,000 does *not* include cost of test specimens, nor expenses of committee members incurred in any phase of the test programs; nor does it include cost of publishing reports of the tests, which will as heretofore be met from general funds of the Society.

Excellent progress has been made in raising the desired funds. About $50,-000 has been pledged by the following nineteen companies, who were approached first because of their known interest in and support of ASTM exposure testing:

Allegheny Ludlum Steel Corp.
Aluminum Company of America
Armco Steel Corp.
Bell Telephone Laboratories, Inc.
Bethlehem Steel Co., Inc.
Crucible Steel Company of America
Dow Chemical Co.
E. I. du Pont de Nemours and Co., Inc.
General Electric Co.
Gulf Research and Development Co.
International Nickel Co., Inc.
Jones & Laughlin Steel Corp.
Republic Steel Corp.
Standard Oil Development Co.

Union Carbide and Carbon Corp.
United States Steel Corp.
Vanadium Corporation of America
Westinghouse Electric Corp.
Youngstown Sheet and Tube Co.

Contributions from these companies range from $1000 to $5000, spread over a period of years or given in one sum as the companies preferred. A number of other companies are considering an invitation to contribute.

With this very substantial start, the appeal for financial support is now being extended to several hundred companies including the members of those ASTM committees interested in this type of testing, who find the results of atmospheric deterioration tests of materials of direct value. However, a contribution in support of this project from any member of the Society—whether approached directly or not—will be most welcome, and our members are earnestly invited to consider such contribution. Checks should be drawn to the order of "ASTM Test Site Fund" and mailed to the Executive Secretary of the Society.

The great value to industry of the Society's findings on the properties of materials in resisting corrosion and other forms of deterioration has long been recognized. The cooperative nature of this work, with participation of both producer and consumer interests, as well as the careful planning and critical evaluation of results, has led to a wide acceptance of ASTM data as engineering facts that can be relied upon in design of structures and equipment, and to the development of many authoritative specifications that are very generally used throughout industry. We are confident, therefore, that the Society's enlarged program of exposure testing described in this article will receive the full and whole-hearted support of our ASTM membership, and in confident anticipation of such support the Advisory Committee on Corrosion is proceeding with the program.

Fig. 6.—Test Site at Pittsburgh (Brunot Island), Pa., Nearly Surrounded by Piles of Cinders.
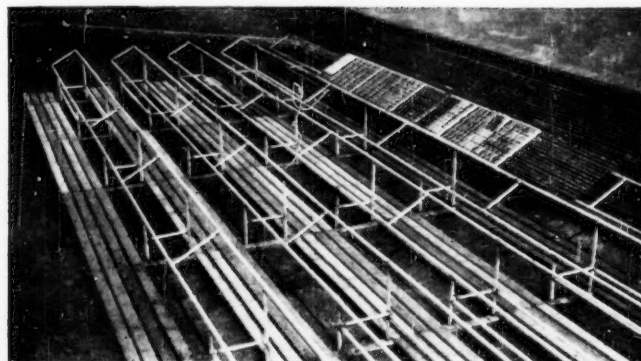
Fig. 7.—Installation on Port of New York Authority Building, New York City.

Standards Committee Approves Many New Tentatives and Changes in Standards

Actions Affect Metals, Alloys, Cement, Masonry, Fuels, Soils, Industrial Water, and Other Materials

THE Administrative Committee on Standards held its usual fall meeting at ASTM Headquarters on September 26. Action was taken on recommendations submitted by many of the Society's technical committees. A detailed list of all the revisions and new additions appears in the accompanying table. These approvals will of course be reflected in the 1950 Supplements to all Six Parts of the 1949 Book of ASTM Standards or in the 1950 Chemical Analysis of Metals, all of which should be available within a few months.

Generally the Administrative Committee on Standards meets but once a year—about two months after the Annual Meeting. It meets more often if there is need for such a meeting. (Many members may recall the special meeting of the Administrative Committee on Standards during the Annual Meeting in New York in 1945 when travel restrictions permitted the Society to hold only a General Business Session.) However, it can and does act on committee recommendations at any time during the year by letter.

The Administrative Committee has for its functions broadly (1) to promote the standardization work of the Society; (2) to consider means of general policy concerning standardization activities, including relationship with similar activities of other bodies; (3) to consider the desirability of expansion of standardization work into new fields; (4) to review annually the progress in the Society's standardization work; and (5) to pass upon proposed new standards, proposed amendments of existing tentatives, or proposed amendments of existing standards offered between annual meetings of the Society in accordance with provisions of the Regulations Governing Technical Committees.

In carrying out its broad functions and its specific authority on committee recommendations the committee is interested in improving the editorial preparation and presentation of proposed methods of test and specifications, and has given considerable attention to the subject.

Committee officers presenting recommendations to the Administrative Committee indicate why the recommendations are being made; give a complete analysis of the vote in the committee; explain the negative votes which are quoted verbatim; and advise what action the committee has taken or proposes to take in order to reconcile the negative viewpoint. At its meetings the Administrative Committee on Standards offers an opportunity for those who have negative viewpoints to present their reasons in person, and for committee officers to be heard in support of committee recommendations. Negative voters on committee recommendations have a right to request such an open hearing at any time.

A classification of actions which can be taken by the Administrative Committee for the Society can be summarized as follows:

1. *Acceptance as Tentative:*
   (a) Acceptance of Tentative Revisions of Standards
   (b) Acceptance of New Tentatives
   (c) Acceptance of Revisions of Tentatives
   (d) Acceptance of Revisions of Standards that are being reverted to Tentatives (essentially acceptance of a new replacing Tentative with concurrent withdrawal of the Standard)
2. *Withdrawals:*
   (a) Withdrawal of Tentatives
   (b) Withdrawal of Standards
   (c) Withdrawal of Tentative Revisions of Standards

Ferrous Metals

Steel:

Committee A-1 on Steel has included all of its boiler steel plate general requirements in a new document (A 20). Its format is identical with that of the A 16 specifications covering structural steel; the latter had been received by producers and consumers alike with such enthusiasm that Committee A-1 decided to revise the boiler steel plate specifications along the same lines. The specifications whose *general requirements* have been taken out and included in the new A 20 are the following:

Specifications for:

Boiler and Firebox Steel for Locomotives (A 30).
Carbon-Silicon Steel Plates for Boilers—Intermediate Tensile Range (A 201).
Nickel-Steel Plates for Boilers (A 203).
Molybdenum-Steel Plates for Boilers (A 204).
High-Tensile-Strength Carbon-Silicon Steel Plates—Plates 6 in. and Under in Thickness (A 212).

Manganese-Vanadium Steel Plates for Boilers (A 225).
Carbon-Steel Plates of Flange Quality—Low - and Intermediate - Tensile Strength (A 285).
Carbon-Manganese-Silicon Steel Plates for Boilers—High Tensile Strength (A 299).
Steel Plates for Pressure Vessels—Low-Temperature Service (A 300).
Chromium-Molybdenum Steel Plates for Boilers (A 301).
Manganese-Molybdenum Steel Plates for Boilers (A 302).

Also the Specifications for Open-Hearth Iron Plates (A 129) and those for Chromium - Manganese - Silicon Alloy - Steel Plates for Boilers (A 202) were revised and reverted to tentative, taking out the *general requirements* which will be included in the new A 20 document.

All of the above specifications, the specific requirements of which are now presented in much more condensed form than previously, will now refer to A 20 concerning the general requirements. These recommendations were approved by the Standards Committee.

The Standards Committee also approved a recommendation by Committee A-1 which brings the Specifications for Heat-Treated Alloy-Steel Bars (A 286) up to date with present commercial practice. There has also been some editorial rearrangement.

In compliance with many requests received to add grades of steel with lower mechanical requirements than in the present specifications for carbon steel bars subject to mechanical property requirements (A 306), Committee A-1 has revised the old standard.

The Standards Committee also approved a recommendation by Committee A-1 which will revise the Tentative Specifications for Hot-Rolled Alloy-Steel Bars (A 322) so that they will cover grades 5160, 8615, 8660, and 9840. Again, requirements for these grades were added to the specification to satisfy many requests.

To complete the series of specifications which Committee A-1 has been writing for the past four years to cover the field of steel bars for ASTM, a new Tentative Specification for Cold Finished Alloy-Steel Bars was approved.

Non-Ferrous Metals

Light Metals:

The Standards Committee approved a recommendation of Committee B-7 on Light Metals and Alloys, Cast and Wrought, that the chemical limits for casting alloys be expressed only to the first

Actions by the ASTM Administrative Committee on Standards, September, 1950

New Tentatives

Specifications for:

General Requirements for Delivery of Rolled Steel Plates of Flange and Firebox Qualities (A 20 – 50 T)

Cold-Finished Alloy-Steel Bars (A 331 – 50 T)

Quicklime for Calcium Carbide Manufacture (C 258 – 50 T)

Hydrated Lime for Grease Manufacture (C 259 – 50 T)

Air-Entraining Admixtures for Concrete (C 260 – 50 T)

Crushed Stone, Crushed Slag, and Gravel for Single Bituminous Surface Treatment (D 1139 – 50 T)

Substitute Ocean Water (D 1141 – 50 T)

Methods for:

Chemical Analysis of Magnesium Sulfate (Epsom Salts) Technical Grade, $MgSO_4.7H_2O$ (C 244 – 50 T)

Analysis of Magnesium Chloride ($MgCl_2.6H_2O$) (C 245 – 50 T)

Testing Magnesia (MgO) for Magnesium Oxychloride Cements (C 246 – 50 T)

Determination of Ignition Loss and Active Calcium Oxide in Magnesium Oxide for Use in Magnesium Oxychloride Cements (C 247 – 50 T)

Slump Test for Field Consistency of Magnesium Oxychloride Cements (C 249 – 50 T)

Mixing Oxychloride Cement Compositions with Gaging Solution for Preparation of Specimens for Laboratory Tests (C 251 – 50 T)

Determination of Linear Contraction of Magnesium Oxychloride Cements (C 252 – 50 T)

Determination of Linear Change of Magnesium Oxychloride Cements (C 253 – 50 T)

Determination of Setting Time of Magnesium Oxychloride Cements (C 254 – 50 T)

Determination of the Consistency of Magnesium Oxychloride Cements by Means of a Flow Table (C 255 – 50 T)

Chemical Analysis of Nickel-Copper Alloys (E 76 – 50 T)

Chemical Analysis of Antimony Metal (E 86 – 50 T)

Sampling Non-Ferrous Metals and Alloys in Cast Form for Determination of Chemical Composition (E 88 – 50 T)

Methods of Test for:

Bulk Density of Magnesium Oxychloride Cement Compositions (C 248 – 50 T)

Field Determination of the Specific Gravity of Gaging Solutions of Magnesium Chloride and Magnesium Chloride-Magnesium Sulfate for Magnesium Oxychloride Cement Compositions (C 250 – 50 T)

Transverse Strength of Magnesium Oxychloride Cement Compositions Using Simple Bar with Knife Edge or Two-Point Loading (C 256 – 50 T)

Compressive Strength of Magnesium Oxychloride Cement Compositions (C 257 – 50 T)

Analysis of Natural Gases by the Volumetric Chemical Method (D 1136 – 50 T)

Analysis of Natural Gases and Related Types of Gaseous Mixtures by the Mass Spectrometer (D 1137 – 50 T)

Resistance to Plastic Flow of Fine-Aggregate Bituminous Mixtures (D 1138 – 50 T)

Amount of Material Finer than No. 200 Sieve for Soil (D 1140 – 50 T)

Recommended Practice for:

The Laboratory Measurement of Airborne Sound Transmission Loss of Building Floors and Walls (E 90 – 50 T)

Methods of:

Chemical Analysis for Aluminum in Zinc Base Die-Casting Alloys (E 47 – 50 T)

Chemical Analysis for Sulfur in Special Brasses and Bronzes (E 54 – 50 T)

Estimation of Average Ferrite Grain Size in Steels (E 89 – 50 T)

Photometric Methods for:

Chemical Analysis of Lead, Tin, Antimony and Their Alloys (E 87 – 50 T)

Tentative Revisions of Standards

Specifications for:

Structural Clay Load-Bearing Wall Tile (C 34 – 49)

Concrete Building Brick (C 55 – 37)

Structural Clay Non-Load-Bearing Tile (C 56 – 49)

Structural Clay Floor Tile (C 57 – 49)

Hollow Load-Bearing Concrete Masonry Units (C 90 – 44)

Hollow Non-Load-Bearing Concrete Masonry Units (C 129 – 39)

Solid Load-Bearing Concrete Masonry Units (C 145 – 40)

Portland Cement (C 150 – 49)

ASTM Thermometers (E 1 – 50)

Methods of:

Sampling and Testing Concrete Masonry Units (C 129 – 39)

Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30 – 47)

Revision of Standard and Reversion to Tentative

Specifications for:

Open-Hearth Iron Plates of Flange Quality (A 129 – 47)

Chromium-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels (A 202 – 47)

Drain Tile (C 4 – 24)

Methods of Test for:

Brinell Hardness of Metallic Materials (E 10 – 27)

Chemical Analysis of Pig Lead (E 37 – 45)

Chemical Analysis of Lead- and Tin-Base Solder Metal (E 46 – 49)

Chemical Analysis of White Metal Bearing Alloys (E 57 – 49)

Revisions of Tentatives

Specifications for:

Boiler and Firebox Steel for Locomotives (A 30 – 49 T)

Carbon-Silicon Steel Plates of Intermediate Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 – 49 T)

Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 – 49 T)

Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 – 49 T)

High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (Plates 6 In. and Under in Thickness) (A 212 – 49 T)

Manganese-Vanadium Steel Plates for Boilers and Other Pressure Vessels (A 225 – 49 T)

Low and Intermediate Tensile Strength Carbon-Steel Plates of Flange and Firebox Qualities (Plates 2 in. and Under in Thickness) (A 285 – 49 T)

Heat-Treated Alloy-Steel Bars (A 286 – 46 T)

High Tensile Strength Carbon-Manganese-Silicon Steel Plates for Boilers and Other Pressure Vessels (A 299 – 49 T)

Steel Plates for Pressure Vessels for Service at Low Temperatures (A 300 – 47 T)

Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 301 – 49 T)

Manganese-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 302 – 49 T)

Carbon-Steel Bars Subject to Mechanical Property Requirements (A 306 – 47 T)

Hot-Rolled Alloy-Steel Bars (A 322 – 49 T)

Aluminum-base Alloy Sand Castings (B 26 – 50 T)

Aluminum-Base Alloy Permanent Mold Castings (B 108 – 50 T)

Aluminum and Aluminum-Alloy Sheet and Plate for Use in Pressure Vessels (B 178 – 50 T)

Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 50 T)

Standard Strength Clay Sewer Pipe (C 13 – 44 T)

Glazed Masonry Units (C 126 – 44 T)

Air Entraining Portland Cement (C 175 – 48 T)

Extra-Strength Clay Pipe (C 200 – 44 T)

Structural Clay Facing Tile (C 212 – 49 T)

Methods of:

Test for Air Content of Air-Entraining Portland Cement Mortar (C 185 – 49 T)

Testing Air-Entraining Admixtures for Concrete (C 233 – 49 T)

Test for Specific Gravity of Soils (D 854 – 45 T)

Chemical Analysis of Aluminum and Aluminum-Base Alloys (E 34 – 49 T)

Methods for:

Chemical Analysis of Copper-Nickel and Copper-Nickel-Zinc Alloys (E 75 – 49 T)

Photometric Methods for:

Chemical Analysis of Magnesium and Magnesium-Base Alloys (E 61 – 46T)

Chemical Analysis of Copper and Copper-Base Alloys (E 62 – 46 T)

Determination of Iron in Slab Zinc (Spelter) (E 64 – 46 T)

Recommended Practice for:

Photometric Methods for Chemical Analysis of Metals (E 60 – 46 T)

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decimal place unless the figure in the second place should be "5" for technical reasons. Committee B-7 also proposed a revision of the Tentative Specifications for Aluminum-Base Alloy Permanent Mold Castings (B 108). Again, this revision will result in expression of chemical limits to the first decimal place unless the second place figure is a "5."

In order to make the Tentative Specifications for Aluminum and Aluminum-Alloy Sheet and Plate for Use in Pressure Vessels (B 178) acceptable to the ASME Boiler Code Committee, the Standards Committee has approved the B-7 recommendation to add requirements for alloys MG11A and G1A and also some data on such properties and characteristics as temper, thickness, tensile strength, elongation, and yield strength.

Certain other necessary revisions which had been overlooked and not included in the 1950 Report of Committee B-7 were submitted to the Administrative Committee for approval. One of these revisions (Table I) changed the zinc limit for alloy ZG32a to "2.7–3.3 per cent." The other revision in the table changes the limit for alloy ZG61a to show zinc as "5.2–6.0 per cent"—Titanium "0.1–0.25 per cent"—and Total Others as "0.2 per cent max."

Cement and Cementitious Materials

Magnesium Oxychloride Cements:

The Administrative Committee on Standards approved twelve separate recommendations of Committee C-2 on Magnesium Oxychloride Cements in the form of new tentative methods. Since approximately 50 million square feet of oxychloride cement flooring and marine decking are installed annually, there was ample justification of the demand by architects, engineers, contractors, and testing laboratories for accredited methods of test for the required materials. These new tentatives approved by the Standards Committee are methods for analyzing magnesium sulfate and magnesium chloride, methods for testing magnesia for use in magnesium oxychloride cement, methods for determining ignition loss and active calcium oxide in magnesium oxide, method for determining slump and field consistency of magnesium oxychloride cement, and a field test method for determining specific gravity of gaging solutions of magnesium chloride and magnesium chloride-magnesium sulfate for the oxychloride cement compositions. A new tentative method for mixing oxychloride cement compositions with gaging solution for laboratory test specimens, one test for bulk density of magnesium oxychloride compositions, a method for determining linear contraction of the cement, a method for finding setting time, a determination of linear change of magnesium oxychloride cement, a method for determining consistency by the flow table, a transverse strength test of magnesium oxychloride cement composition using simple bar with knife edge or 2-point loading, and last, but not least, a compression strength test for magnesium oxychloride cement compositions.

Clay Pipe:

Committee C-4 on Clay Pipe had submitted to the Standards Committee several recommendations.

To relieve the unnecessary restriction which is now placed upon size of socket and to eliminate the problem which had been created previously whenever the precast type of jointing compound is used, the Tentative Specifications for Standard Strength Clay Sewer Pipe (C 13) have been revised. Also increased manufacture of longer lengths of pipe in the larger diameters necessitated a change to the longer lengths of fittings. These same comments apply to the Specifications for Extra-Strength Clay Pipe (C 200) which have also been similarly revised.

Lime and Concrete:

Two new tentatives submitted by Committee C-7 on Lime were approved: The Specifications for Quicklime for Calcium Carbide Manufacture and Specifications for Hydrated Lime for Grease Manufacture had been developed to guide the consumers in standardizing their requirements for lime.

Committee C-9 on Concrete and Concrete Aggregates recommended new tentative Specifications for Air-Entraining Admixtures for Concrete. It is highly important to have specifications which will permit the differentiation among the large numbers of air-entraining admixtures which have appeared on the market today—permitting determination of those that are and those that are not suitable. A companion Tentative Method of Testing Air-Entraining Admixtures for Concrete (C 233) which had been issued earlier has also been revised to bring it up to date.

Masonry Units:

Committee C-15 on Manufactured Masonry Units recommended a number of revisions of specifications. The first of these, on Structural Clay Facing Tile (C 212), was revised to increase tolerances on permissible distortion since former tolerances were too restricted. The second, on Glazed Masonry Units (C 126), was revised to provide for a more simplified arrangement of description of units. Refinements in the sections on absorption requirements and color and texture have been made. Changes in sizes of single-face units (Table II of the specification) are made to conform to masonry practice as affected by the mortar thicknesses. This also applies to tolerances on dimensions. Further refinement was made in the section on coring to conform with similar changes in the section on description of unit.

The new revision of the Specifications for Hollow Non-Load-Bearing Concrete Masonry Units (C 129) included an additional source of aggregate and thus recognizes new material suitable for use. References to modular sizing have also been added.

A revision of the Standard Specifications for Concrete Building Brick (C 50) also recognizes new materials suitable for use. In addition, since present practice and ex-

perience has indicated that higher minimum compressive strengths are reasonable, requirements for Grade D brick have been revised. In the light of present practice and experience, modulus of rupture requirements are not considered necessary in the use of brick and masonry and therefore they have been deleted in this revision. The water absorption properties and mixture control sections have had control limits added. Committee C-15 also submitted a revision of Standard Specifications for Hollow Load-Bearing Concrete Masonry Units (C 90) to include an additional source of aggregate.

The Standards Committee approved revisions of the Specifications for Structural Clay Floor Tile (C 57), Structural Clay Load-Bearing Wall Tile (C 34), and Structural Clay Non-Load-Bearing Tile (C 56). An additional paragraph was added to provide requirements for maximum average weight of closed-end floor units.

To secure more uniformity of test results and as a refinement of the method, the Standards Committee also approved a revision of the Methods of Sampling and Testing Concrete Masonry Units (C 140). The specifications for Solid Load-Bearing Concrete Masonry Units (C 145) has also been revised to conform with Specifications C 90. A complete revision of the standard specification for Drain Tile (C 4) was submitted as the first since 1924.

Cement:

The revision of Tentative Specifications for Air-Entraining Portland Cement (C 175) provides the long-desired substitute for the requirement that Committee C-1 test additions, or review results of tests of additions, in order to provide a list of acceptable additions by a reference to the new standard, C 226 - 50 T.

Miscellaneous Materials

Gaseous Fuels:

Committee D-3 on Gaseous Fuels submitted two new tentative methods. The one for analysis of natural gas and related gaseous mixtures by the mass spectrometer was developed by the committee as part of a major program covering a number of methods. Since favorable results were obtained using the mass spectrometer for gas analysis a method was formulated based on this instrument's use. The other new tentative method is a Volumetric Chemical Method. Again, after much study and experiment on various methods which have been employed for the determination of chemical composition of gaseous fuels this method was formulated as one which would be suitable as a standard.

Road and Paving Materials:

Committee D-4 on Road and Paving Materials submitted a new tentative method for plastic flow resistance of fine-aggregate bituminous mixtures. The basis of the method has been commonly known as the Hubbard-Field Stability Test; it has been widely used for many years in evaluation of bituminous paving mixtures comprising mineral aggregates of small size. Since it is the method that is

generally used, it should be standardized. It is one of a number under study in Committee D-4, one other of which has already been published as a Compressive Strength Test for Bituminous Mixtures (D 1074).

The Standards Committee, in approving a tentative Specification for Crushed Stone, Crushed Slag, and Gravel for Single Bituminous Surface Treatment, has acted on the first standard that the Society has developed on mineral aggregates for use in bituminous surface treatment of highway and similar services. This specification will supplement the group for mineral aggregates which now includes D 692, D 693, D 694, and D 1073.

Soils:

Committee D-18 proposed a revision of the Tentative Test Method for Specific Gravity of Soils (D 854). The present revision brings the method up to date.

A new method of test for the amount of material finer than No. 200 sieve in soils was approved by the Standards Committee, as no standard tests have been heretofore recommended on this subject and since there is a recognized need for such a standard.

Simulated Ocean Water:

The Standards Committee approved a new tentative specification for substitute ocean water. This specification was developed by Committee D-19 on Industrial Water at the request of the Advisory Committee on Corrosion. Committee D-2 had originally requested such a specification be developed in order that they might use it in their corrosion testing of oil and lubricants.

General Subjects

Thermometers:

A revision of the Standard for ASTM Thermometers, E-1 includes detailed specifications for 18 new precision thermometers. Many test methods are of such a nature that use could be made of a large type precision thermometer available from most manufacturers rather than to set up separate thermometer specifications for individual methods. The thermometer subcommittee prepared for such use these specifications for a set of 18 fractionally graduated test thermometers (9 in Fahrenheit and 9 in corresponding Centigrade ranges).

Hardness of Metals:

A revision of the standard test for Brinell hardness of metallic materials has also been approved. The revision will provide for use of the 1500-kg. load for testing metals of intermediate hardness in addition to the present loads previously covered. Recommendations have been added for use of the several loads for various hardness ranges. Recommendations on types of balls, load-time indentations, space indentation, curved surface testing, indentation measurement accuracy, Brinell permanent deformation resistance, and apparatus calibration. Also added is an appendix in the form of a table showing minimum thickness of specimens required for Brinell hardness tests.

Chemical Analysis of Metals:

The Administrative Committee on Standards also acted on five new tentatives, four revisions of tentatives, a tentative revision of one standard, and revisions of three standards recommended for reversion to tentative, these items being held over by Committee E-3 which had not found it possible to include them as part of its annual report. The methods for Chemical Analysis of Nickel-Copper Alloys and those for Chemical Analysis for Sulfur in Special Brasses are in effect tentative revisions of the Standard Method of Chemical Analysis of Special Brasses and Bronzes (E 54). Photometric Methods for the Chemical Analysis of Antimony, the Methods for the Chemical Analysis of Lead Tin, Antimony and their alloys and those for the Chemical Analysis for Aluminum in Zinc-Base Die-Casting Alloys are in effect revisions of the Standard Methods of Chemical Analysis of Zinc-Base Die-Casting Alloys (E 47). The tentative recommended Practice for Photometric Methods for Chemical Analysis of Metals (E 60) and the tentative Photometric Method for the Determination of Boron in Steel (E 30) have been revised by adding new methods for determining beryllium and tin in steel. A procedure for the determination of bismuth and several changes in the old procedures have been included in Photometric Methods of Analysis of Aluminum Alloys (E 34). Addition of new methods for determining copper and nickel in magnesium base alloys has been made to Photometric Methods for Chemical Analysis of Magnesium and Magnesium Base Alloys (E 51). A new method for determining silicon in copper-base alloys has been added to the tentative Photometric Methods for Chemical Analysis of Copper and Copper-Base Alloys (E 62).

Committee E-3 had also submitted revisions of standards (to be reverted to tentative) on Chemical Analysis Pig Lead (E 37), to include the former Photometric Method for the Determination of Bismuth in Pig Lead (E 58). The Chemical Analysis of Lead and Tin-Base Solder Metal (E 46), and Chemical Analysis of White Bearing Metal (E 57), were also revised and reverted to tentative. A new tentative, Method of Sampling Non-Ferrous Metals and Alloys in Cast Form for Chemical Analysis, was also submitted by Committee E-3. This method will cover the general principles of samples applicable to the non-ferrous metals and is not intended to supersede or replace existing specifications or recommendations for sampling a particular material.

The ferric chloride and salicylate methods for determination of iron have been deleted from the Photometric Methods for Determination of Iron in Slab Zinc (Spelter) (E 64).

A tentative salicylate method for analyzing copper nickel alloys formerly published with designation E 63 has been incorporated in the Tentative Methods of Chemical Analysis of Copper-Nickel and Copper-Nickel-Zinc Alloys (E 75).

Metallography, Building Constructions:

To provide ASTM coverage for ferrite grain size standards, Committee E-4 on Metallography had submitted a Tentative Method of Classifying Ferrite Grain Size in Steels.

In order to define in considerable detail the test method now in current use for measuring the transmission loss of large-scale building walls, floors, and other elements of building construction such as doors and windows, Committee E-6 on Methods of Testing Building Constructions had submitted to the Standards Committee a new tentative test method for laboratory measurement of air-borne sound transmission loss of building floors and walls.

The paragraphs above have given in some detail some technical notes concerning most of the various approvals made by the Administrative Committee on Standards at the September Meeting. Readers may refer to the accompanying table which lists all of the approvals for complete titles and designations of new tentatives and for the latest revisions of existing tentatives and standards.

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1951 Annual Meeting

THE 1951 Annual Meeting of the Society will be held at the Chalfonte-Haddon Hall in Atlantic City the week of June 18 to 22. A large technical program for this meeting is anticipated and the program is now being developed. It is expected that sessions on the following subjects will be held: flame photometry, sandwich constructions, acoustical materials, sampling of bulk materials, consolidation of soils, and surface and subsurface reconnaissance of soils. There will, of course, be many other technical papers presented in addition to the above, covering varied subjects of interest.

Any January, 1950, BULLETINS?

THE demand for the January, 1950, ASTM BULLETIN has far exceeded expectations and, at the present time, the remaining stock is practically nil. Since there will be some need here at Headquarters for this particular issue we are particularly anxious to recall some of those which were sent to members and friends. All members who feel that they have no further need for this particular issue and who feel they would be willing to part with it may return it to Headquarters. The Society will pay 50 cents to anyone returning this issue of the BULLETIN.

NEW ASTM PUBLICATIONS

SEVERAL important AS-TM additions to materials testing literature have been published recently or will be available within a few weeks.

A Report on Standard Samples for Spectrochemical Analysis, the 1950 Yearbook, Supplements to the Metal Cleaning Bibliographical Abstracts and Electrical Contacts, the new edition of Chemical Analysis of Metals, and an Index for the X-Ray Diffraction Cards are some in these categories. In the paragraphs which follow brief descriptions of the various books and pamphlets mentioned above and others are given.

Index for Revised X-Ray Diffraction Data Card File Soon Available

IN 1950 the Society published for the Joint Committee on Chemical Analysis by X-Ray Diffraction Methods a revised card file comprising upward of 4000 X-ray diffraction patterns with related information. This information was furnished either on plain cards or cards of the "Keysort" variety as requested by the purchasers.

Now at the printers and expected to be available around the end of October is the index to the card file. The index is being printed in two sections in one cloth book, one section being a grouped numerical index, the other an alphabetical index. For ease of reference the two sections will be printed on paper of different colors.

The numeric index will contain listings for three variations of the three strongest lines in each pattern as follows:

first, second, third
second, first, third
third, first, second

The alphabetical index will contain listings for several permutations of the chemical name with its elements and radicals so that this index may be utilized for locating a given compound in the card file.

The book will total approximately 700 pages and will be distributed free of charge to all purchasers of the card file, one to each purchaser.

1950 Year Book Published

THERE is in course of distribution, to each ASTM member who has requested a copy, the 1950 Year Book. This 580-page publication gives much information which is of constant service to the members. About 150 pages are devoted to an alphabetic list of the membership, with the addresses and titles of the individual members and the company representatives shown. One of the most interesting and most useful portions (over 250 pages) of the Year Book is the committee personnel section. This section lists all technical and other committees and their officers and gives the complete personnel of the main committees and subgroups. Anyone not cognizant of the work of the Society would no doubt be amazed at the large number of committees and at the number of men and organizations serving on them.

Other portions of the Year Book are devoted to the By-laws and Charter, and various sets of regulations, lists of winners of awards, information on administrative committees and District Councils and (Just a reminder!) there are bound in the book ASTM membership application blanks so that members will have these available when a new membership is in the offing.

This book is distributed to members on request and many have asked that their name be added to the permanent mailing list. Each new member receives a copy and has the opportunity of asking that his name be added to the permanent mailing list.

The Year Book is not available for general distribution and is published for distribution to the members for their use in connection with the activities of the Society.

Electrical Contacts

A REVISED and expanded 1949 Supplement (20 per cent larger than last year) to the Bibliography and Abstracts on Electrical Contacts includes most of the current articles on electrical contacts which have been published. The present Supplement becomes the sixth to the original Bibliography and is presented as a service to the interested workers of this field by the following members of Subcommittee X on Electrical Contacts of Committee B-4 on Electrical Heating, Resistance, and Related Alloys:

E. I. Shobert II, Stackpole Carbon Co., *Chairman*
George Durst, General Plate Division of Metals and Controls, Inc.
V. E. Heil, P. R. Mallory & Co.
C. K. Strobel, Robertshaw Thermostat Corp.

The price is 75 cents; members can obtain the Supplement for 60 cents. The original book with all supplements is $6.75.

Chemical Analysis of Metals

INTERESTED members are urged to be on the lookout for the 1950 edition of the Chemical Analysis of Metals. The greatly revised and expanded book will be the first new edition since 1946 and will continue to cover chemical methods for analysis of steel, cast iron, open hearth iron, and wrought iron, and all of the ferro alloys and non-ferrous metals and alloys—aluminum, magnesium, copper, brass, bronze, bearing metal, lead, tin, nickel, zinc heating alloys, and die casting alloys. Very soon a prospectus will be mailed to members and others interested in the fields covered by the Book, describing in detail this important A.S.T.M. collection of standards. An order blank will be included. The cloth-bound book of about 500 pages will be $6.50. Price to A.S.T.M. members will be $4.50

Metal Cleaning

THE 1950 Supplement to A.S.T.M. *STP No. 90* on Metal Cleaning Bibliographical Abstracts by J. C. Harris brings up to date the coverage by this bibliography of the published data on metal cleaning. References arranged in indexes by author and by year are now covered from 1893 to 1950. In addition to the author index, there are subject, specification, and patent indexes to facilitate reference to the abstracts. Wherever possible, the original articles have been abstracted, but in some cases articles are re-abstracts from journals such as *Chemical Abstracts* or *Journal of the Institute of Metals*.

Interested users are urged to send the author references that should be added and corrections that should be made. This information will be included in the next amplification of the bibliography. The Supplement is $1.00 and price to members is $.75; The original book is $2.75; with 1950 supplement, $3.00.

Spectrochemical Analysis Samples

THE results of a new survey are published to provide, in a form for ready reference, current information on spectrochemical standard samples (*STP No. 58B*). The results of this survey supplement the information

which was offered in a 1943 report (by W. R. Brode and B. F. Scribner) and a revision of the latter in 1947.

The rapid growth of spectrochemical analysis and a corresponding increase in available standard samples make a periodic compilation such as this, of types and sources of standards, of extreme value to analysts. As a result of this current survey conducted by Alan Goldblatt of Committee E-2 on Emission Spectroscopy, the Section on Pure Substances has been expanded to a list of 325 entries supplied by 43 sources. The total number of entries of standard samples and pure substances in this report is 1077. The report is $1.25. The price to members is 90 cents.

1950 Supplements to the Book of Standards

WORK HAS already been started to prepare the 1950 Supplements to the current 1949 Book of ASTM Standards, looking toward the publication of these Supplements, to come out at varying intervals over the period November 30–February 1. There will be a Supplement to *each* of the six Parts, bound in heavy paper cover.

Exactly when each Supplement will appear has not been determined but it is probable that those for the Parts 2, 4, and 6 covering Non-Ferrous Materials; Paint, Wood, Paper, etc.; and Electrical Insulation, Plastics and Rubber, respectively, may appear first.

Each *member* will receive automatically the Supplements matching the Parts of the Book of ASTM Standards he has already received, these instructions being on file at Headquarters. All *purchasers* of the Book of Standards, including the many members who buy extra Parts, will receive in the next few weeks an order form by which they can purchase the Supplements to match the Books of Standards they have bought.

With the Supplements will be stickers noting changes in standards so that members can affix these at the designated pages in the big books and thus aid in keeping these up to date. Obviously the respective Supplements should be placed right next to the Parts of the big book so that the latest standards can be referred to conveniently. The Supplements will contain not only revised material but also many new specifications and tests.

Soon after the last Supplement is issued there will be printed another Index to Standards which will give the latest references to the appropriate source where the specifications can be found. This is sent to all members and each purchaser of the Book.

Textile Compilation Now Available

CONTAINING all the standards on textile materials and related information on photomicrographs of fibers, a yarn number conversion table, humidity table, three technical papers on stress-strain problems and committee data, the new compilation of ASTM Standards on Textile Materials covers most of the widely used products of this industry.

The current revision of this compilation continues to provide testing methods, acceptable tolerances, and specification requirements on quality. This 1950 edition, it is hoped, will provide conveniently all the data and information believed to be of importance to those who deal with textile materials.

An important part of the compilation is the glossary of textile terms and terms related to hand of fabrics.

Many general standards are included on such things as snag resistance, fire-retardant properties, air permeability, interlaboratory testing, insect pest damage resistance of pile floor coverings, water resistance, microorganism resistance, and light fastness. There are also many standards on cotton, rayon and silk, hosiery, and wool.

The 600-page (84 standards) compilation developed by Committee D-13 on Textile Materials and containing a detailed index is $4.50. ASTM members can buy the compilation for the special price of $3.40.

A Correction

AN ERROR has been noted in the Year Book which has just come off press. The personnel of the Administrative Committee on Research appearing on page 2 is in error in that it lists J. W. Bolton as still a member of the committee although, as indicated, his term expired in 1950, and the name of L. C. Beard, Jr., has been omitted instead and should appear.

Members will no doubt wish to note this correction in their copy of the Year Book.

Errata in Heger-Miller Report

J. J. HEGER, co-author of the Report on the Strength of Wrought Steels at Elevated Temperatures, has advised Society Headquarters of the following errors in the Report:

On page 9, References for Carbon Steel, reference 11 should read as follows:
(11) A. E. White, C. L. Clark, and R. L. Wilson, "Influence of Carbon Content on High-Temperature Properties of Steels *Transactions*, Am. Soc. Metals, Vol. 23, p. 995 (1935).

Add a reference 19 to the same page:
(19) A. E. White, C. L. Clark, and R. L. Wilson, "Influence of Time at 1000 F. on the Characteristics of Carbon Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, p. 139 (1936).

The curve for rupture strength *versus* temperature of the 2.25 per cent chromium - 1.0 per cent molybdenum on page 4 should be shifted to the right 100 F. In other words, the rupture strength value shown for a temperature of 900 F. is actually the rupture strength at 1000 F.

Reprints of Evans Paper Available

IN VIEW of the many inquiries for the paper by C. T. Evans, Jr., on "Oil Ash Corrosion of Metals at Elevated Temperatures" which was presented as part of the Symposium on Corrosion and Erosion of Gas Turbine Materials, it is proposed to make reprints available. The paper is of widespread interest and all those wishing to secure copies of the paper should advise Headquarters as promptly as possible. The price will be 50 cents.

Offers of Papers for 1951

THE Administrative Committee on Papers and Publications will meet early in February to consider the papers to be published by the Society in 1951 and to develop the program for the 1951 Annual Meeting to be held in Atlantic City, N. J., June 18–22. All those who have in mind offering papers for presentation at the Annual Meeting and publication by the Society should send these offers to Society Headquarters *no later than January 16*. All offers should be accompanied by a Summary which should make clear the intended scope of the paper and indicate features that, in the opinion of the author, will justify its inclusion in the Annual Meeting program and publication by the Society. Suitable blanks to be used in transmitting the desired information will be sent promptly on request.

The American Standards Association and the ASTM

EDITOR'S NOTE.—Frequent reference is made from time to time in the ASTM BULLETIN and in the Report of the Board of Directors and of its administrative committees, to the activities of the American Standards Association—more recently particularly in connection with work being undertaken with the International Organization for Standardization (ISO). It is accordingly felt appropriate that a more extended statement appear in these pages concerning the American Standards Association and the relationship of ASTM to the ASA.

THE American Standards Association (ASA) is a federation with over 100 technical and professional societies, trade associations, and consumer organizations constituting the member bodies and it serves as a clearing house for standards activities in this country and internationally.

The American Standards Association by its name and history epitomizes the democratic approach to standardization. Standards arise as the voluntary and cooperative effort of all those concerned in any given field to arrive at agreement on what is good practice. These standards develop in many fields of activity and are channeled through trade, consumer, and technical association. The democratic coordination designed to arrive at American Standards is the task of ASA. In much of this work ASTM cooperates and in some of it takes a leading part.

To accomplish this coordination, procedures and methods have been established to insure the development of a consistent set of American Standards, so essential to the American economy both in normal times and in periods of national strain. The actual formulation of the standards rests for the most part with committees organized for the purpose by the sponsoring groups.

The procedures set up are truly democratic in the sense that every group substantially concerned with a standard is guaranteed the right to participate in deciding what the provisions of the standard shall be—so that the standard shall represent a national consensus. Decisions are to be arrived at not by simple majority vote but, rather, by the consensus principle; threshing out most points so thoroughly that any decision reached is unanimous or nearly so. Minorities are protected, special interests tempered with public interest, and national acceptance assured for American Standards.

The judicial character of these procedures offers a framework of rights and privileges which provide equal opportunity to all groups to advance the national economy through voluntary American Standards.

Founded as the American Engineering Standards Committee in 1918, during the earlier World War, by five engineering societies, including the ASTM, to provide a forum where organizations interested in standards could meet on a common basis, its broadened membership works today:

1. To simplify the development of engineering, commercial, consumer, and safety standards;
2. To eliminate duplication, overlapping, and variations in standards activities among groups developing standards in the U. S.—all looking toward a single, generally accepted American Standard;
3. To promote the knowledge and use of standards as tools of management and as an element of American industry, business and technology; and
4. To serve as a focus for standards work on an international basis.

Neither a trade association nor a technical society, the ASA is a service organization—a piece of machinery—through the use of which standards or ideas for standards may be coordinated. The ASA does not in any sense compete with its members but functions rather to avoid any duplication of projects. As developed by its members to coordinate and channel their standards activities cooperatively, it also serves other organizations, which frequently bring standards problems to the ASA for coordination.

Relation of ASTM and ASA:

As mentioned above, the ASTM was one of the founders of the ASA and is one of its most active members. It makes use of the ASA facilities in various ways—in securing recognition of many of the ASTM standards as "American Standard," as discussed below, and in some few instances carries out the actual development of standards directly under ASA procedure. In organizing new ASTM committees, the Society has the cooperation of the ASA in developing and securing a truly representative personnel, and in having these committees coordinated with other work in the field.

In view of its very broad interest in the activities of the ASA, it is only natural that the ASTM should play an active part in its administration.

The management of ASA is vested in the Board of Directors consisting of representatives of member bodies nominated by the Standards Council. The ASTM has had such representation over the years in the membership of the Board of Directors by direct nomination, as follows:

Quincy Bent—1929–1936
G. W. Thompson—1938–1939
Jerome Strauss—1939–1943
H. H. Morgan—1945–1950

F. M. Farmer, one of the ASTM representatives on Standards Council, served as ex-officio member of the Board of Directors by reason of his office as Chairman and Past-Chairman of Standards Council. The Society also took a very active part in the affairs of the American Engineering Standards Committee, the predecessor of the ASA, through the active participation of A. A. Stevenson, J. A. Capp, A. W. Gibbs, and F. M. Farmer.

Responsibility for standardization activities in ASA rests with the Standards Council, on which the ASTM is presently represented by J. R. Townsend and C. H. Rose, with J. H. Foote and R. E. Hess as alternates, Mr. Townsend being Vice-Chairman of the Council as well as Chairman of the Board of Review, an important committee through which much of the work of the Council clears. The work of Standards Council is subdivided and placed in the hands of a number of so-called correlating committees, to supervise the activities in specific fields as indicated below. These are made up of representatives of the member bodies at work in these fields and in a sense are small standard councils. On most of them the ASTM is represented as indicated:

Electrical Standards Committee—F. M. Farmer
   The committee has two subdivisions—on both of which Mr. Farmer represents the ASTM: Power Division and Communications and Electronics Division
Mechanical Standards Committee—J. R. Townsend (Vice-Chairman)
Mining Standardization Correlating Committee
Building Code and Construction Standards Correlating Committee—Miles N. Clair (Member of its Executive Committee)
Consumer Goods Committee—A. G. Ashcroft
Safety Code Correlating Committee—Myron Park Davis (Member of its Executive Committee)
Chemical Industry Correlating Committee—H. K. Nason
Miscellaneous Projects Correlating Committee—G. H. Harnden (Chairman)

The procedure of the ASA provides for the development and recognition of standards in various ways. Two meth-

ods are currently employed by ASTM in submitting standards to the ASA. One that is particularly appropriate for ASTM standards is the so-called existing standards procedure under which an existing ASTM standard is submitted with a supporting statement giving a history of the development of the standard in ASTM and indicating the degree of its acceptance by industry. If the standard is approved as American Standard, the ASTM is granted proprietary sponsorship to provide for its future revisions, and such revisions are brought to the attention of the ASA currently for approval under the proprietary sponsorship procedure.

The second method employed by ASTM consists of having standards reviewed in a sectional committee made up of representatives of a number of interested organizations but with ASTM as sponsor or co-sponsor for the sectional committee. Fifteen sectional committees are now functioning this way, as follows:

A1 on Specifications and Methods of Test for Hydraulic Cements

A2 on Specifications for Fire Tests of Materials and Construction (Jointly with the Fire Protection Group and National Bureau of Standards)

A21 on Specifications for Cast-Iron Pipe and Fittings (Jointly with Am. Gas Assn., Am. Water Works Assn., and New England Water Works Assn.)

A37 on Road and Paving Materials

A42 on Specifications for Plastering (Jointly with the Am. Inst. of Architects)

A88 on Magnesium Oxychloride Cement Flooring (Jointly with the National Bureau of Standards)

B36 on Standardization of Dimensions and Materials of Wrought-Iron and Wrought-Steel Pipe and Tubing (Jointly with the Am. Soc. of Mechanical Engineers)

C7 on Bare Electrical Conductors (formerly Sectional Committee H4)

C59 on Electrical Insulating Materials

J6 on Specifications for Protective Equipment for Electrical Workers (Jointly with the Edison Electric Inst.)

L3 on Specifications for Rubber-Lined Fire Hose (Jointly with ASA Fire Protection Group)

L14 on Textile Test Methods (Jointly with Am. Assn. of Textile Chemists and Colorists)

O4 on Methods of Testing Wood (Jointly with U. S. Forest Service)

Z11 on Petroleum Products and Lubricants

Z23 on Specifications for Sieves for Testing Purposes (Jointly with National Bureau of Standards)

At one time there was a Sectional Committee G8 on Galvanized Iron and Steel and a Sectional Committee M20 on Classification of Coal but the responsi-

bility for any standards in these fields have been assigned to ASTM Committees A-5 and D-5, respectively. The Sectional Committee on Drain Tile (A6) has recently been disbanded and responsibility for any standards in the field of drain tile has been delegated to ASTM Committee C-15.

Approximately 150 ASTM standards have approval as "American Standard" under the existing standards procedure, and approximately 215 ASTM standards have been approved as American Standard on the recommendation of the sectional committees.

The Society keeps in touch with the work of a number of other sectional committees functioning under the ASA by having official representation. There are 39 such sectional committees on which the Society's interest is represented as indicated on page 532 of the 1950 ASTM Year Book.

The American Standards Association provides the American contact with the International Organziation for Standardization (ISO); in fact, the ASA is the American member of the ISO. The ASTM is interested in a number of these international projects as follows:

Textiles	Viscosity
Petroleum	Paper
Rubber	Iron and Steel
Solid Mineral Fuels	Chemistry
Plastics	Copper and Copper Alloys

Shellac (A subcommittee under ASTM Committee D-1 functions as the American committee)

Mica (A subcommittee under ASTM Committee D-9 functions as the American committee)

Insulating Oil (A subcommittee under Committee D-9 functions as advisory to the U. S. Committee of the International Electrotechnical Commission)

General Definitions Relating to Chemical and Physical Tests

It will be seen from the above that the Society places considerable value and importance on the work of ASA, for it looks upon the ASA as a help in integrating the standardization activities of the ASTM into the over-all standardization work of American Industry. It is felt that the Society has contributed much from its experience to the operations of the ASA.

Instrument Industry May Face War Manpower Shortage

THE current recall of reserve officers and enlisted personnel to the armed forces, and to a lesser extent the draft, has brought into the open and emphasized a critical national shortage of manpower in certain highly skilled classifications.

In an attempt to solve the problem, representatives of the Society for Apparatus Manufacturers of America have been working in Washington to establish classifications and quotas for critical instrument specialists and procedures for their deferment. Industry representatives met early in September in Washington to discuss this problem and the establishment of priorities of instrument materials.

Schedule of ASTM Meetings

DATE	GROUP	PLACE
October 18–20	Committee D-13 on Textile Materials	New York, N. Y.
October 19–20	Committee D-14 on Adhesives	Boston, Mass.
October 19–20	Committee D-15 on Engine Antifreezes	New York, N. Y.
October 23–25	Committee C-16 on Thermal Insulating Materials	Asbury Park, N. J.
October 26–27	Committee B-4 on Electrical Heating, Resistance, and Related Alloys	Philadelphia, Pa.
October 26–27	Committee C-19 on Structural Sandwich Construction	Madison, Wis.
November 6	Pittsburgh District	Pittsburgh, Pa.
November 8	New England District	Cambridge, Mass.
November 13	Committee C-20 on Acoustical Materials	Boston, Mass.
November 14–15	Committee D-20 on Plastics	Cincinnati, Ohio
November 15–17	Committee D-9 on Electrical Insulating Materials	Cincinnati, Ohio
November 30	Philadelphia District	Philadelphia, Pa.
November 30	California District	Los Angeles, Calif.
January 15–16	Board of Directors	Philadelphia, Pa.
January 16	Philadelphia District	Philadelphia, Pa.
March 5–9	SPRING MEETING AND COMMITTEE WEEK	Cincinnati, Ohio
June 18–22	ANNUAL MEETING	Atlantic City, N. J.
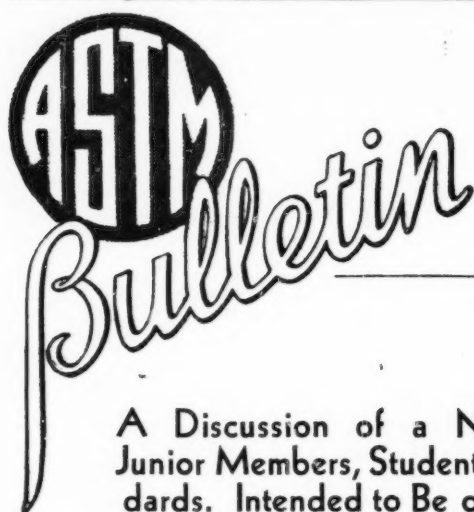
A Discussion of a New Book, Student and Junior Members, Student Prize Awards, Use of Standards. Intended to Be of Interest to Every Member

Board of Directors Anxious to Stimulate Junior Membership—New Textbook Pertinent in this Regard

RECENTLY in perusing a new and significant student textbook on "Materials of Construction" by John H. Bateman, an ASTM member and Professor of Civil Engineering, Louisiana State University, it appeared that we might well refer to certain portions of this text or use excerpts therefrom, and convey with these the desire of the ASTM Board of Directors to stress the significance in its operations of a continually growing student and junior membership. If in so doing we may indirectly present something of a review of the new publication this may be somewhat of a "give-and-take" proposition because Professor Bateman, in our opinion, has incorporated remarkably well in his new text many fine tributes to the work of ASTM. How he does this will be, we hope, apparent from the material below.

At its September meeting the Board of Directors, in suggesting that a study be made of how the number of junior members might be increased, reiterated its feeling that a knowledge of ASTM, especially what work it does, and how that work is carried out, should be very beneficial to any engineering student who is going to be concerned with materials. The corollary also was stressed that the more widespread is the knowledge of ASTM among students and young engineers, the more widespread will be the benefits accruing from the intensive work of the Society's technical committees.

Professor Bateman in his Preface has something to say about this situation, as follows:

"The young engineer, upon graduation, may come into contact with materials of construction in a number of ways. He may be involved in the selection of materials in connection with the design of structures. He also may have some part in the preparation of specifications for materials. Prior to and during construction he will be concerned with the inspection of materials, which may include the making of tests of various kinds. As an inspector on construction, he will be required to participate in the fabrication and erection of structures . . . It is therefore essential that he have a background of knowledge of the raw materials, production, and properties of the principal materials of construction . . . Practically every area in the United States and Canada, and in other countries as well, is confronted with problems concerning materials of construction which are peculiar to itself. . . The student should have a reasonably good knowledge of available sources of information about materials of construction upon completion of his course of study. The various industries and engineering organizations connected with construction are constantly revealing new knowledge of materials. The publications presenting new information are numerous, and the student will find his efforts rewarded if he will acquaint himself with the work and publications of such organizations as the American Society for Testing Materials. . . ."

The author then lists other societies, all of which are cooperating in various phases of ASTM work.

The Society attempts to provide every facility so that the engineering student member can become acquainted with our work. The dues are kept very low—$2 per year—and the policy with respect to furnishing publications is liberal. These include the BULLETIN, the Year Book, Index to Standards, and the special Compilation of Selected Standards for Students—all for the $2 dues. If the student wants any one of the special compilations, for example the Standards on Textiles or Petroleum or Cement, he can get these for a very nominal additional charge. The number of student members varies greatly depending somewhat upon our national and economic situation. At the present time there are about 500, but these are heavily concentrated at three or four schools where the schools are taking advantage of the privilege accorded student members to purchase the Book of Standards at greatly reduced prices.

There is being mailed to each member a prospectus describing a Student Membership Prize Award plan which has several objectives: first, to recognize the work of outstanding students; second, to increase the number of student memberships; and it also provides an opportunity for members at a very nominal outlay to underwrite a movement constructive from the standpoint of the Society and the students and their schools.

Standards for Students in Engineering:

For many years the Society has issued a special Compilation of Selected Standards for Students in Engineering, the most recent printing of which is 35,000 copies. This book is furnished to every student member (unless he wishes to secure one of the special compilations of standards instead). Engineering schools, not only in this country but in other parts of the world, use this 300-page book in the laboratory and with other texts, particularly in courses involving the testing of materials. In this connection Professor Bateman notes: "the student's understanding of materials of construction and their adaptation to engineering uses will be much more complete if a course of study includes laboratory courses in the physical testing of materials and in the mechanics of concrete mixtures."

All Items on Standards Ballot Approved

THE canvass of the results of the 1950 letter ballot shows that the membership of the Society has approved all of the items listed on the ballot. The list included one adoption of a new standard, 92 revisions of existing standards, and 58 adoptions of tentatives as standards. Details concerning the actions were given in the Summary of Proceedings which was mailed to all members in August, and they will be published in the 1950 Supplements to the 1949 Book of A.S.T.M. Standards which are now in preparation.

Junior Membership:

Quite naturally there stems from any discussion of student membership a consideration of what the Society does to facilitate the student becoming a full member of ASTM. One means which has been available for many years is the Junior Membership class. This provides a convenient transition for the student eventually to become a full member of the Society. The dues for Junior Membership, $9, are half the regular dues of $18, and the entrance fee of $5 likewise is half the regular fee. A Junior Member has all the privileges of a full membership except that he cannot hold office. (Some of our hard-working officers may perhaps sigh for the good old days when they were Junior Members!) Junior Membership should be of more widespread interest than it has been. Now there are more than 100 Junior Members, and the Board believes this number should be increased and has taken steps to study the matter. Whether the present entrance fee of $5 is a deterrent has been discussed. Any young person can be a Junior Member until the beginning of the fiscal year next succeeding his 27th birthday; then he must assume the status of an Individual Member.

References to ASTM Standards:

The Board's Developmental Committee in considering the policy with respect to permission to reprint ASTM standards did not reach a decision on all points and is continuing this study, but it is recommending that where permission to reprint is given we ask that it be emphasized that the ASTM standards may be revised from time to time, and to note that for the official source and the latest information the actual specifications of the Society should be consulted. In his new text Professor Bateman has some pertinent words for the user of his engineering text, to wit:

"Numerous references to the *Standards* of the American Society for Testing Materials and the American Association of State Highway Officials are contained in this text. It should be understood that these, and other, standards are modified and revised at frequent intervals. Every effort has been made to refer to the standards in effect at the time of completion of the manuscript but changes may be in effect by the time of completion of printing."

New Book on "Materials of Construction":

After using material from the preface to the new book "Materials of Construction" by John H. Bateman, issued by the Pitman Publishing Corp., 2 West 45th St., New York, N. Y., and available at $6.50 per copy, we would be unappreciative if we did not give a few more details about the publication, which we are glad to do. First, it may be stated that this is not the first good text that has been issued for students of engineering, nor will it probably be the last. A number of other texts previously issued have been reviewed in the ASTM BULLETIN, and we are glad to bring pertinent publications of this kind to the attention of our readers. A list of chapter headings will convey the coverage of Professor Bateman's publications; these are:

The pages shown indicate the amount of material the author devotes to each chapter.

Several things about the publication impress us, for example there is a very extensive bibliography of what seem to be current and up-to-date references at the end of each chapter. (Many of the papers and publications are ASTM.) These bibliographies should be extremely valuable to any engineering student. Professor Bateman did not ignore the responsibility of including a rather detailed index and this should be of real student value.

We would rather expect that the author of any text on materials would find it desirable to incorporate many references to ASTM, and Professor Bateman has been prolific in his use of ASTM material, all references, whether to ASTM or other bodies, being carefully tied in with suitable footnotes.

Being partial, we particularly like the section on "Questions and Problems" at the end of each chapter—for example "What tests are applied to . . . sewer pipes? (Consult current ASTM Standards.)" Or again: "Tabulate the physical-test requirements of the ASTM Specifications for Paving Brick," or "How do woods fail when subjected to direct compression loads parallel to the grain? (See ASTM Methods D 143.)" Or "What is the range of tensile strength of steel for bridges and buildings, and for structural rivets? (Consult ASTM Standards.)"

We believe this new book is an outstanding publication. Any engineering student who spends some time conscientiously with the publication will have an excellent broad-range knowledge of materials, and particularly will be able, when he gets out into industry, or wherever he may be, to go to pertinent sources of current information. The book is well printed with numerous illustrations.

The 1951 Nominating Committee

IN ACCORDANCE with the By-laws providing that the Board of Directors shall select a nominating committee for officers, the Board has considered the report of the tellers—E. J. Albert, Thwing-Albert Instrument Co., and C. R. Hutchcroft, Keasbey and Mattison Co.—on the recommendation of members for appointees on the nominating committee and alternates, and has appointed the following:

Nominating Committee

Members

H. C. Larson, Bethlehem Steel Co., Inc.
K. G. Mackenzie, The Texas Company
W. C. Hanna, California Portland Cement Co.
J. R. Townsend, Bell Telephone Laboratories, Inc.
H. L. Maxwell, E. I. du Pont de Nemours and Co., Inc.
F. G. Steinebach, *The Foundry*, Penton Publishing Co.

Respective Alternates

J. W. Price, Jr., Carnegie-Illinois Steel Corp.
W. T. Gunn, American Petroleum Institute
R. E. Davis, University of California
G. R. Gohn, Bell Telephone Laboratories, Inc.
W. T. Pearce, Consulting Engineer
Sam Tour, Sam Tour and Co., Inc.

Serving on the 1951 Nominating Committee as *ex-officio* members are the three immediate past-presidents: T. A. Boyd, Richard L. Templin, and J. G. Morrow. The committee will meet sometime in March and nominate for each office—president, vice-president, and five members of the Board of Directors. The selections by the Nominating Committee will be announced to the members in the ASTM BULLETIN prior to transmission of official ballots.

Another Case History on Use of Standards in Research

Standards Aid in Corrosion Prevention Work

THROUGH the close co-operation of one of our active members, Dr. M. H. Brown,[1] Metallurgist, E. I. du Pont de Nemours Co., Inc. and member of the Joint Committee on Filler Metal and Committee A-10 on Iron-Chromium-Nickel and Related Alloys, we are able to present below another in the series of case histories which have stressed the importance of recognized standard test procedures in research work and development of new products. Previous articles have pointed out that in such fields as paint, motor fuels, use of metals in high-temperature service and others, standards have been in a key position in enabling research work to progress.

Those who attended the recent annual meeting will recall a display of photographs illustrating the series of articles which were published in previous BULLETINS. Many of the photographs showed products which have resulted from research work where standard tests were used.

For many years, A.S.T.M. has carried out important research and standardization activities involving corrosion-resistant or so-called stainless steels, and Dr. Brown's discussion is therefore pertinent.

This exploring of the significance of standards in research is part of a more extensive project which, among other aims, is intended to impress management, as well as the technical fraternity, with a better appreciation of the significance of A.S.T.M. work.

Boiling Nitric Acid Test for Corrosion-Resistant Steels

By M. H. Brown[1]

The A.S.T.M. Recommended Practice A 262 – 44 T describes a corrosion test in boiling 65 per cent nitric acid which is widely employed for the family of alloys known as the stainless steels. Quoting directly from the document, this test "has been found to be a very useful and reliable method of measuring a quality of corrosion-resisting steels, especially with regard to the effective-

ness of heat treatment. It is used primarily as a quality control and inspection test and has also been employed to advantage in research leading to improvements in composition, manufacture, and treatment of corrosion-resisting steels."

The purpose of this case history is to show why the test was originally developed, how it has been used, and to point out its advantages and limitations. It is now commonly referred to as the boiling 65 per cent nitric acid test, the boiling nitric acid test, or merely the nitric acid test, but is also still widely known as the Huey test, after its originator, W. R. Huey. The test was first described by Huey in 1930[2] and was the subject of an A.S.T.M. Subcommittee report in 1933.[3]

In the early days of stainless steel (only about twenty years ago), these alloys were installed in many services involving exposure to solutions containing nitric acid, which previously have been handled in nonmetallic containers or in high-silicon iron. However, numerous mysterious corrosion failures of stainless equipment occurred which were found, upon investigation, to be related to the heat treatment of the material and which for the austenitic alloys were predominantly intergranular in nature. It was also found that improperly heat-treated material could be detected by subjecting a representative specimen to a corrosion test in boiling 65 per cent nitric acid, thus permitting corrective measures to be taken prior to installation of equipment. The first use of the boiling 65 per cent nitric acid test might thus be considered as a simulated service test. As the use of the stainless alloys was expanded, however, it was found that many other service environments which did not contain nitric acid also attacked improperly heat-treated material intergranularly. The boiling nitric acid test proved to be a reliable criterion for detecting susceptibility to intergranular corrosion (note the restriction) not only to nitric acid but to these other solutions as well. The function of the test as routinely employed thus became that of comparing an unknown (the material represented by the specimen under test) with a standard (the typical

performance of properly heat-treated material) to determine whether or not the unknown was susceptible to intergranular corrosion. Over a period of time the method became quite widely used for this purpose. The necessity for meeting this standard unquestionably played a major role in improving the quality of stainless steel through, for instance, improved heat-treating practice and control of carbon content. The test also became an important research tool in work aimed at the improvement of the stainless alloys. A recent appraisal of the test in comparison with other evaluation methods was given by Brown, DeLong, and Myers.[4]

A great deal of misunderstanding regarding the scope of the boiling nitric acid test, persisting over the years, was primarily responsible for delaying issuance of an A.S.T.M. Recommended Practice until 1944. The impression has apparently been held by some that the test was or could be used to determine general corrosion resistance to any and all media. This is definitely not the case. So far as general corrosion resistance is concerned, results of the test are applicable only to boiling 65 per cent nitric acid. Neither are they applicable for predicting whether or not pitting or stress-corrosion cracking will occur in other media. The primary purpose of the test in routine evaluation is to detect susceptibility to intergranular corrosion. In this sense, and *only* in this sense, the results are applicable to any media which will intergranularly corrode susceptible material. However, the test does not indicate *which* other media are capable of attacking susceptible material in this manner, or how quickly. Such information can only be gained through prolonged exposure in the particular media involved or by service experience. There are also certain other possible limitations, as pointed out below. However, the test has proved itself extremely valuable when properly employed and has contributed greatly to reducing intergranular failures in service from the status of a common occurrence to almost a rarity.

For the unstabilized alloys of conventional carbon contents, correlation

[2] W. R. Huey, "Corrosion Test for Research and Inspection of Alloys," *Transactions*, Am. Soc. Steel Treating, Vol. 18, p. 1126 (1930).

[3] "Report of Subcommittee IV on Methods of Corrosion Testing," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 187 (1933).

[4] M. H. Brown, W. B. DeLong, and W. R. Myers, "An Appraisal of Methods for Evaluating the Corrosion Resistance of Stainless Steels," Symposium on Evaluation Tests for Stainless Steels, *STP No. 93*, Am. Soc. Testing Mats. (1949).

of boiling nitric acid test data with service experience in the manner indicated above has been amply demonstrated. For type 304 the results seem to be directly related to carbide precipitation. For types 316 or 317, where phases other than austenite are more likely to be present, the latter may also contribute to increased corrosion rates on improperly heat-treated material, but carbide precipitation is still the controlling factor. Secondary phases probably also contribute to increased rates, perhaps sometimes importantly, in the case of the stabilized alloys. With the new extra-low-carbon type 316 grade (0.03 per cent carbon max.), however, recent work indicates that increased corrosion rates in the boiling nitric acid test as the result of exposure in the unfavorable temperature range may be due principally to sigma phase rather than to carbide precipitation. If this is true, it remains still to be proved for ELC 316 whether or not other media will intergranularly corrode material indicated to be susceptible by the boiling nitric acid test, as they do its higher carbon counterpart, where carbide precipitation is the controlling factor. The existence or absence of such a relationship can only be proved by long-time exposure tests or by operating experience. No similar complications are indicated to apply to ELC 304, for which the test seems definitely applicable.

It is probable that boiling nitric acid test requirements have sometimes been enforced when the service media in which the stainless steel was to be employed was not capable of intergranularly corroding even material which had been shown to be susceptible to that form of attack. Many service environments fall into a borderline class in which it is uncertain whether or not susceptible material will be so attacked. A field corrosion testing program intended to develop specific information on this point is being sponsored by the High Alloys Committee of the Welding Research Council. Both the A.S.T.M. and the National Association of Corrosion Engrs. are cooperating. In this program, properly and improperly heat-treated specimens of various stainless alloys, together with as-welded, and welded-and-stress-relieved specimens of the same materials, are to be exposed to a wide variety of service media. Evaluations representing the various conditions will be made not only by the boiling nitric acid test, but by two other methods employed for the same purpose—the boiling acidified copper sulfate test and the nitric-hydrofluoric acid test.

At the present time consideration is being given to an alternate method of conducting the boiling nitric acid test as proposed by DeLong,[5] which involves the use of apparatus for exposing a large number of specimens in a single container. Data are being accumulated to establish whether A.S.T.M. A 262 - 44 T should be revised to permit the alternate procedure.

ASTM DISTRICT ACTIVITIES

Meetings—Current and Future

New York (Effects of Atomic Bombing)
Ohio Valley (Air and Water Pollution)

Pittsburgh (President's Smoker)
New England (Wood and Wood Products)

Philadelphia (Conservation)

As this BULLETIN is being prepared, interesting meetings are imminent in several ASTM Districts. In New York, on October 6, Edward J. Kehoe, Chief, Fire and Accident Branch, New York Operations Office, U. S. Atomic Energy Commission, is to speak on "Effects of Atomic Bombing."

In Columbus, under the auspices of the Ohio Valley District, is a potentially very interesting meeting on "Air and Water Pollution" with, as chief speakers, L. F. Warrick, Chief, Technical Services Branch, Division Water Pollution, U. S. Public Health Service, and H. G. Dyktor, Commissioner, Division Air Pollution Control, City of Cleveland.

Meetings in Pittsburgh, Boston, and Philadelphia:

President L. J. Markwardt will visit two of the Districts in November. On Monday, November 6, he will be the guest at a Smoker and President's Night at the Pittsburgh University Club. Then, on Wednesday, November 8, the New England District is planning a technical meeting with President Markwardt as the featured technical speaker. He will present interesting information on wood and

⁵ W. B. DeLong, "Testing Multiple Specimens of Stainless Steels in a Modified Boiling Nitric Acid Test Apparatus," Symposium on Evaluation Tests for Stainless Steels, *STP No. 93*, Am. Soc. Testing Mats. (1949).

wood products, including the remarkable sandwich constructions and composites being developed which incorporate many other materials such as metals, textiles, and adhesives.

In Philadelphia, on November 30, at the Franklin Institute, several speakers will discuss various aspects of conservation of our natural resources such as forests and waters, including renewable resources.

Further details of all these meetings will be sent by direct mail to all members in the respective Districts.

1951 Award of Merit Committee Appointed

ACTING under the rules governing the A.S.T.M. Award of Merit, President Markwardt, with the approval of the Board of Directors, has appointed a committee which will review suggestions for awards to be given in 1951. Past-President Richard L. Templin will represent the Board of Directors on this committee, and the two hold-over members from the previous group will be L. H. Winkler and Carlton H. Rose. Two new members will be appointed for two-year terms. Mr. Winkler has been designated Chairman of the current Award Committee.

Although the Award of Merit Committee may on its own initiative make nominations to the Board of Directors for men to receive the Award of Merit, essentially it is a review group to consider suggestions received from the A.S.T.M. technical committees. The rules provide that these awards which recognize outstanding contributions and leadership in A.S.T.M. work—in particular, technical activities—are to be distributed among the several general committee groups. All suggestions from the technical committees must be in the hands of the Award of Merit Committee not later than February 1. The officers of technical committees have received a communication concerning the award with copies of the latest rules, and the latter are also published in the 1950 A.S.T.M. Year Book.

The first of these new Awards of Merit were given by the Board of Directors at the 1950 Annual Meeting. The July BULLETIN included the citations for the various men as well as their photographs.

Performance Tests to Be Stressed in Development of Wax Polish Standards

PERFORMANCE standards will occupy a prominent place in the initial program of the newly formed Committee D-21 on Wax Polishes and Related Materials. The first meeting since organization was marked by a sincere desire of those present to organize and begin the task of assembling the necessary background of data required before the actual formulation of the much-needed performance and chemical and physical property standards in the field can go forward. A two-day meeting was held on September 14 and 15 at the National Bureau of Standards, Washington, D. C. During this time each subcommittee held its first meeting and developed its organization. Following the subcommittee meetings the main committee meeting was held at which time proposed By-laws were adopted and election of permanent officers was held. The officers elected for the term ending at the Annual Meeting of the Society in 1952 are Chairman, W. W. Walton, National Bureau of Standards; Vice-Chairman, J. V. Steinle, S. C. Johnson and Son Co.; and Secretary, B. S. Johnson, Franklin Research Co.

The business of the subcommittees consisted essentially of formulating a scope of activities and adopting initial programs of work. The Subcommittee on Nomenclature will limit its activities to the definitions and descriptions of terms used in the manufacture and evaluation of wax polishes and related materials necessary to the proper functioning of the committee. Two comprehensive terms which will receive initial consideration are "wax polish" and "other related materials." The scope of the Subcommittee on Raw Materials concerns the preparation of test methods and specifications for raw materials entering into the manufacture of wax polishes and related materials. The first objective will include a review of methods published by the American Wax Importers Association. A survey of raw materials now used will be made. The Subcommittee on Physical and Chemical Tests, after careful consideration, adopted a scope embodying research and development of physical and chemical test methods for the evaluation of finished wax polishes and related materials prior to their use or application. Existing appropriate ASTM test methods as well as other sources will be listed and reviewed for their adaptation to the work of the committee. Physical property determinations such as pH, total solids, heat stability, and freezing characteristics will be developed into ASTM standards. Even though it is expected that the committee will not be in a position to adopt specifications for some time, pending the development of test methods, the subcommittee on specifications set up a scope which will include the development and preparation of specifications for finished wax polishes and related materials.

Performance tests apparently will be one of the fields of endeavor of most interest to the committee and the wax polish industry as evidenced by the many requests for active participation in the Subcommittee on Performance Tests. The scope of this subcommittee as adopted includes research and development of procedures for testing the performance of wax polishes and related materials. The magnitude of work to be covered in this subcommittee has warranted the formation of particular sections to work on specific performance projects. These sections will be: Section A on Slip Resistance, Section B on Wear, and Section C on Appearance. Much valuable discussion took place on this very pertinent subject of performance in which it was brought out that there are many factors which will be involved in developing slip resistance tests. Decision was reached to use linoleum as a standard flooring sample, and arrangements were made to distribute samples from one specific source to a number of cooperating members for round-robin test work in their laboratories. An interesting talk on "Wax Research" was given by Dr. Steinle before an open meeting to which other than committee members were invited. The talk described in a very interesting manner the development of natural wax resources and refining methods instituted in Brazil and the development of new sources from sugar cane. The next meeting of the committee is planned for Chicago with tentative dates of April 25 and 26, 1951.

Fall Meeting of Ceramic Whiteware Committee

COMMITTEE C-21 Ceramic Whiteware met at Bedford Springs, Pa., on September 21, this meeting preceding the annual fall meeting of the Whitewares Division of The American Ceramic Society. Reports were made indicating fine progress on the part of the Subcommittees on Nomenclature, Tests and Specifications, and Research. Papers presented by the latter group and other papers discussed at the meeting included proposed methods for testing whiteware clays, particle-size determinations, glaze stress, autoclave tests for delayed crazing, smoothness of glaze surfaces, tensile testing at elevated temperatures, determination of absorption, and outline of methods of phase identification for whiteware bodies. The over-all progress of this relatively new committee has been most satisfactory.

Calendar of Society Meetings

"Long" and "short" calenders will appear in alternate BULLETINS. The "short" calendar notes meetings in the few immediate weeks ahead—the "long" calendar for months ahead.

SOCIETY FOR APPLIED SPECTROSCOPY—November 7, Socony-Vacuum Training Center, New York, N. Y.

NATIONAL FOUNDRY ASSOCIATION—Annual Meeting, November 8–10, Edgewater Beach Hotel, Chicago, Ill.

FEDERATION OF PAINT AND VARNISH PRODUCTION CLUBS—28th Annual Convention, November 9–11, Congress Hotel, Chicago, Ill.

AMERICAN PETROLEUM INSTITUTE—Annual Meeting, November 13–16, Biltmore & Ambassador Hotels, Los Angeles, Calif.

NATIONAL PAINT, VARNISH AND LACQUER ASSOCIATION—Convention (Federation Day), November 17–18, Fairmont Hotel, San Francisco, Calif.

AMERICAN STANDARDS ASSOCIATION—Annual Meeting, November 27–29, New York, N. Y.

AMERICAN SOCIETY OF MECHANICAL ENGINEERS—Annual Meeting, November 27–December 1, Hotel Statler, New York, N. Y.

AMERICAN INSTITUTE OF CHEMICAL ENGINEERS—Annual Meeting, December 3–6, Neal House, Columbus, Ohio.

AMERICAN ASSOCIATION FOR THE ADVANCEMENT OF SCIENCE—117th Meeting, December 26–31, Public Auditorium and Downtown Hotels, Cleveland, Ohio.

SOCIETY OF AUTOMOTIVE ENGINEERS—Annual Meeting & Engineering Display, January 8–12, 1951, Book-Cadillac, Detroit, Mich.

SOCIETY OF PLASTICS ENGINEERS, INC.—7th Annual National Technical Conference, January 18–20, 1951, Hotel Statler, New York, N. Y.

AMERICAN INSTITUTE OF MINING & METALLURGICAL ENGINEERS—Annual Meeting, February 18–22, 1951, Jefferson Hotel, St. Louis, Mo.

American Society for Testing Materials—Spring Meeting and Committee Week, March 5–9, 1951, Netherlands-Plaza Hotel, Cincinnati, Ohio.

7TH WESTERN METAL CONGRESS & EXPOSITION—March 19–23, 1951, Oakland, Calif.

AMERICAN SOCIETY OF MECHANICAL ENGINEERS—Spring Meeting, April 2–5, 1951, Atlanta, Ga.

THE AMERICAN CERAMIC SOCIETY—53rd Annual Meeting, April 22–26, 1951, Palmer House, Chicago, Ill.

PERSONALS...

News items concerning the activities of our members will be welcomed for inclusion in this column.

NOTE—*These "Personals" are arranged in order of alphabetical sequence of the names. Frequently two or more members may be referred to in the same note, in which case the first one named is used as a key letter. It is believed that this arrangement will facilitate reference to the news about members.*

E. Bruce Ashcraft has returned, after a 4-year absence, to the Westinghouse Research Laboratories, Pittsburgh, as Advisory Engineer for the Chemical Dept. He had been serving as Manager of Chemistry Research for the Westinghouse Atomic Power Division.

Bani R. Banerjee, formerly Research Engineer, Standard Oil Co. (Ind.), Chicago has joined the faculty of the Bengal Engineering College, University of Calcutta, India.

Charles S. Barrett, of the Institute for the Study of Metals of the University of Chicago, has been named by The Franklin Institute to receive its Francis J. Clamer Medal for meritorious achievement in metallurgy, the accompanying citation reading: "In recognition of his outstanding contribution to the knowledge of crystal structures and of the effects of deformation and aging on various metallic crystals."

Oliver P. Beckwith has been named Assistant Director of Quality Control, Alexander Smith & Sons Carpet Co., Yonkers, N. Y. Formerly Chief of the company's Product Engineering Laboratories since 1945, Mr. Beckwith joined Smith in 1935 as a Product Engineer. During the latter stages of the recent World War he served as principal textile technologist in the planning division of the Office of the Quartermaster General, U. S. Army, and is currently Chairman of the Pile Fabrics Panel of the Subcommittee on Quartermaster Problems of the National Research Council. Active in A.S.T.M. technical committee work for many years, Mr. Beckwith is Secretary of Committee E-11 on Quality Control and is the present Chairman of the Section on Wool of Committee D-13.

Edward H. Berger, Asphalt Consultant for the Johns-Manville Corp., was inducted recently into the company's Quarter Century Club at a dinner at the Belmont Plaza Hotel, New York City. Mr. Berger is recognized as one of the country's outstanding authorities on asphalts, tars, and pitches.

William J. Edgar, formerly Assistant Director of Laboratories, Federal-Mogul Corp., Detroit, Mich., is now associated with Charles C. Kawin Co., Chicago, Ill.

Leon V. Foster, formerly in charge of Bausch & Lomb Optical Co.'s Scientific Bureau's Microscope Development Section, has been promoted to head of the Military Engineering Dept. of the company. A graduate of Syracuse University, Mr. Foster joined the optical firm in 1917.

Jeff S. Fox has opened his own office as Consulting Engineer in Decatur, Tex. He was previously with the Civil Aeronautics Administration, Fort Worth.

A. P. Gloeckler, formerly Standards Engineer on the central staff of Avco Mfg. Co., is now Chief Metallurgist at the New Idea Div. of Avco at Coldwater, Ohio.

K. W. Graybill, Chief Engineer, Automatic Electric Co., Chicago, Ill., has been elected Vice-President of the company. Mr. Graybill will also remain in the capacity of Chief Engineer, and will continue to direct research, development, and patents.

Roger C. Griffin has retired as Vice-President of Arthur D. Little, Inc., Cambridge, Mass. Mr. Griffin had represented his company in A.S.T.M. for many years, being a member of Committee D-6 on Paper since 1937 and serving as chairman of that group for three years. He was a member of the A.S.T.M. Board of Directors in 1940–1942.

William F. Little, Engineer in Charge, Photometric Dept. of the Electrical Testing Laboratories, Inc., New York City, recently received the 1950 Illuminating Engineering Society Medal, the highest honor in the lighting field, presentation being made at the Opening Session of the Society's National Technical Conference in Pasadena, Calif. The accompanying citation referred to "his nearly half-century of achievement in the development of equipment and standards for measurement and testing of light" and "his outstanding influence in the development of certification programs for the lighting industry."

A. D. Macintyre, Assistant Engineer, Pacific Gas and Electric Co., San Francisco, has retired. Mr. Macintyre had been active in the work of A.S.T.M. Committee C-1 on Cement for several years.

Gerald H. Mains, Development Engineer, National Vulcanized Fibre Co., Wilmington, Del., and **Albert G. H. Dietz,** Associate Professor of Building Engineering and Construction, Massachusetts Institute of Technology, Cambridge, are to be judges in connection with the 1950 Prize Paper Contest of the Society of Plastic Engineers, Inc.—Mr. Mains to be Chief Judge. Full information about the contest may be obtained from the Society of Plastic Engineers, 409 Security Bank Building, Athens, Ohio.

Henry A. Meyners, formerly Chief Chemist, Eastern Tar Products, Baltimore, Md., is now associated with the Mid-West Tar Products Corp., Hammond, Ind., in a similar capacity.

F. C. Moriarty has been named Assistant Vice-President, Aurora Gasoline Co., Detroit, Mich. He was formerly with the Universal Oil Products Co., Chicago.

G. G. Oberfell, Vice-President of Research and Development of Phillips Petroleum Co., Bartlesville, Okla., has retired from active duty under the company's retirement plan.

Albert S. Orr, formerly General Foreman of the Refinery Technology Division (and Assistant to **Herschel G. Smith,** Manager), Gulf Oil Corp., Philadelphia Pa., has been appointed Director of the Refinery Technology Laboratory, following the retirement of Mr. Smith. **T. L. Cantrell** has been named Assistant Director.

C. G. A. Rosen, Director of Research Caterpillar Tractor Co., Peoria, Ill., is James Clayton Lecturer for 1950. The Clayton Lectures, sponsored by the British Institution of Mechanical Engineers, are designed to promote the advancement of knowledge in fields related to mechanical engineering, and are delivered annually by engineers or scientists of world reputation. Mr. Rosen will present the 1950 Lecture on November 14 in London; his subject will be "Significant Contributions of the Diesel Research Laboratory."

L. P. Seyb, since 1942 a chemist and group leader in the Research Department of Diamond Alkali Co., Cleveland, Ohio, has been named Manager of Research.

Angus G. Sturrock has been appointed Manager of the Metallurgical Division of Wyckoff Steel Co., Ambridge, Pa. He has been with the company for 22 years.

The **Universal-Rundle Corp.,** New Castle, Pa., recently dedicated its new $300,000 W. Keith McAfee Laboratory, one of the first and largest of its kind in the plumbing fixture industry. The ultramodern research laboratory was named in honor of Universal-Rundle's late chairman of the board of directors, **W. Keith McAfee** for many years a nationally prominent figure in the ceramic and engineering professions. Dr. McAfee was a former member of A.S.T.M. Committee C-21 on Ceramic Whitewares. **Dr. A. L. Johnson,** director of the new laboratory, is active on Committee C-21, as well as on Committee C-22 on Porcelain Enamel.

Fred Wilhelmy, formerly with The Ansonia O & C Co., Ansonia, Conn., is now associated with the United Elastic Corp., Lowell, Mass., as a designer.

James C. Zeder, formerly Chairman of the Chrysler Engineering Board, has been made Director of Engineering and Research, Chrysler Corp., Detroit, Mich. Mr. Zeder, who first joined the Chrysler Corp. when it was organized in 1925, is currently President of the Society of Automotive Engineers.

NEW MEMBERS...

The following 56 members were elected from August 10, 1950 to September 18, 1950, making the total membership 6752 . . . Welcome to ASTM

Note—Names are arranged alphabetically—company members first then individuals

Chicago District

AMERICAN BILDROK CO., W. L. Morrison, Jr., Director, Research and Development, 2001 W. Pershing Rd., Chicago 9, Ill.

KRAFT FOODS CO., Engineering Dept., 500 Peshtigo Court, Chicago 90, Ill.

SOLAR AIRCRAFT CO., Hiram Brown, Chief Metallurgist, Eighteenth and Grand Ave., Des Moines 5, Iowa.

BARDWELL, RICHARD A., Engineer of Tests, Chicago & Eastern Illinois Railroad, Oaklawn Shops, Danville, Ill.

BOSS, KENNETH A., Technical Director and Plant Manager, Mutual Dealers Wholesale, Inc., 2361 Hampden Ave., St. Paul 4, Minn.

CHU, NELSON C., Engineer, MacDonald Engineering Co., 188 W. Randolph St., Chicago 1, Ill.

GLASER, M. A., Vice-President, Midland Industrial Finishes Co., East Water St., Waukegan, Ill.

LEACH, VERNON G., Consulting Engineer and Director of Research Dept., Peabody Coal Co., 231 S. La Salle St., Chicago 4, Ill.

McNELLY, DONALD P., Quality Control Engineer, Fort Wayne Corrugated Paper Co., Hartford City, Ind.

MILWAUKEE, CITY OF, SEWERAGE COMMISSION, Ray D. Leary, Chief Engineer and General Manager, Box 2079, Milwaukee 1, Wis.

REAGEL, FRED S., Assistant to Technical Director, Marquette Cement Manufacturing Co., 20 N. Wacker Dr., Chicago 6, Ill. [J]*

TEPLE, LAWRENCE S., Mechanical Engineer, A. O. Smith Corp., 3533 N. Twenty-seventh St., Milwaukee, Wis. For mail: 8330 N. Whitney Rd., Milwaukee 11, Wis.

WAKEFIELD, ERNEST H., Director and President, Radiation Counter Laboratories, Inc., 1844 W. Twenty-first St., Chicago 8, Ill.

Cleveland District

KNIGHT, MAURICE A., CO., Maurice A. Knight, Jr., Vice-President, 175 Kelly Ave., Akron 9, Ohio.

FAHLMAN, JOHN E., Metallurgical Engineer, The Permold Co., Box 70, Medina, Ohio. [J]

MAIL, I. P., Research Analyst, National Carbon Research Labs., Box 6087, Cleveland 1, Ohio.

Detroit District

MITCHELL & SMITH, Ralph P. Champney, President, 9501 Copland Ave., Detroit 17, Mich.

STITT, J. R., Research Engineer, The R. C. Mahon Co., Box 4666, Detroit 34, Mich.

New England District

BAXTER, J. V., Quality Manager, United Shoe Machinery Corp., Beverly, Mass.

CARROLL, CHARLES W., Vice-President, Haarmann Steel Co., Box 206, Holyoke, Mass. For mail: 72 Essex St., Holyoke, Mass.

New York District

BUSH, GEORGE FRANKLIN, Owner-Director, G. F. Bush Associates, 16 Model Ave., Hopewell, N. J.

FARRAH, GEORGE A., Technical Director, National Container Corp., Long Island City, N. Y.

LANDES, WILLIAM, Owner, Paper and Pulp Testing Labs., 118 E. Twenty-eighth St., New York, N. Y.

SHIMAMOTO, GEORGE G., Executive Officer, Kelly & Gruzen, 80 Fifth Ave., New York 11, N. Y.

* [J] denotes Junior Member.

Northern California District

GLAIVE, A. F., Chief Food and Drug Chemist, State Department of Public Health, University of California, Berkeley, Calif. For mail: 2022 California St., Berkeley 3, Calif.

Ohio Valley District

PRICE BROTHERS CO., Harry S. Price, Jr., Vice-President, 1932 E. Monument Ave., Box 825, Dayton 1, Ohio.

HAUPT, KARL W., Sales Engineer, National Valve and Manufacturing Co., Pittsburgh, Pa. and Atwood & Morrill Co., Salem, Mass. For mail: Room 504, 519 Main St., Cincinnati 2, Ohio.

Philadelphia District

DEGNAN, THOMAS F., Corrosion Engineer, E. I. du Pont de Nemours and Co., Inc., Chambers Works, Penns Grove, N. J.

LEHR, C. B., Vice-President and General Manager, William Boekel and Co., Inc., 509 Vine St., Philadelphia 6, Pa.

RICHTER, SVEN, Consulting Chemical Engineer, 419 W. Johnson St., Philadelphia 44, Pa.

Pittsburgh District

HAVEKOTTE, WALTHER L., Assistant Manager of Research, Firth Sterling Steel and Carbide Corp., McKeesport, Pa.

LABEKA, CHARLES L., Plant Metallurgist, Pittsburgh Steel Co., Monessen, Pa.

St. Louis District

HUBBARD, B. M., Chief Inspector, Wagner Electric Corp., 6400 Plymouth Ave., St. Louis 14, Mo.

THOMPSON, A. PAUL, Director of Research, The Eagle-Picher Co., Box 290, Joplin, Mo.

Southern California District

SMITH, P. A., Chief Engineer, Selwyn-Landers Co., 4709 E. Washington Blvd., Los Angeles 22, Calif.

Washington (D. C.) District

PETERSEN, PERRY H., Structural Research Engineer, Housing and Home Finance Agency, Washington 25, D. C. For mail: 5019 Allandale Rd., Washington 16, D. C.

RUSSELL, WILLIAM A., Research Engineer, Housing and Home Finance Agency, Washington 25, D. C. For mail: 3440 Thirty-ninth St., N. W., Washington 16, D. C.

Western New York-Ontario District

SLATER, N., CO., LTD., F. H. Vercoe, Plant Manager, Sydney St., Hamilton, Ont., Canada.

U. S. and Possessions

DAKOTA LIME AND BRICK CO., L. R. Kennedy, President and General Manager, Box 631, Rapid City, S. Dak.

SOUTHWIRE CO., Roger J. Schoerner, Manager, Carrollton, Ga.

BROWN, WALTER, Chief Design Engineer, The Brewster Co., Inc., P. O. Drawer 1095, Shreveport 80, La. For mail: 922 Kirby Pl., Shreveport, La.

MARTIN, C. C., Sales Manager, The Standard Magnesia Co., Gabbs, Nev.

THOMAS, HUGUENIN, JR., Partner, Thomas & Hutton, Box 1043, Savannah, Ga.

WATTON, GEORGE, Assistant Chief of Field Station, U. S. Department of Commerce, National Bureau of Standards, University of Washington Campus, Seattle 5, Wash. For mail: 3823 W. Willow St., Seattle 6, Wash.

Other than U. S. Possessions

CEMENTOS DEL NORTE, S. A., Edgardo G. Quiroga, Director, Av. Independencia y Héroes del 47, Monterrey, N. L., Mexico.

COMPTOIR FRANCO-BELGE D'EXPORTATION DE TUBES D'ACIER, 64 Rue Pierre-Charron, Paris 8°, France.

IMPERIAL CHEMICAL INDUSTRIES, LTD., DYESTUFFS DIVISION, J. M. Buist, Senior Physicist, Rubber Service Dept., Hexagon House, Blackley, Manchester, England.

LA CONSOLIDADA, S. A., Felipe Soto, Metallurgist, Apartado Postal 81 Bis, Mexico 2, D. F., Mexico.

DEUTSCHER NORMENAUSSCHUSS E. V., Friesenplatz 16, Koln I, Germany.

DU BELLAY, GRIFFON, Ingenieur, Société Electro-Chimie, Pierre-Benite, Rhone, France.

ENGINEERING COLLEGE, DACCA, The Principal, P. O. Ramna, Dacca, East Pakistan.

HOCKING, H. A., Technical Representative, Mond Nickel Co., Ltd., Claridenstr. 25, Zurich, Switzerland.

JAPANESE STANDARDS ASSN., Mamoru Nakamoto, Chief Managing Director, Patent Agency Bldg., 1 Sannencho Chiyodaku, Tokyo, Japan.

KUDVA, V. S., Managing Director, The Canara Workshops Ltd., Kodiyalbail, South India.

LONG, CHARLES, Ingenieur Principal Service General des Recherches, Société Electro-Chimie d'Ugine, 10 Rue du Plat, Lyon 2°, Rhone, France.

LUZ SANDOVAL, MARIA DE LA, Diaz y Nava, S. A., Gabriel Mancera 1139, Colonia del Valle, Mexico, D. F., Mexico.

NECROLOGY...

The death of the following members has been reported

ALLAN W. AINSWORTH, Manager, Tool and Alloy Steels Div., Horace T. Potts Co., Philadelphia, Pa. (August 13, 1950). Member since 1937.

F. E. BASH, Assistant Vice-President, Driver-Harris Co., Harrison, N. J. (September 14, 1950). Representative of Driver-Harris company membership since 1929. (See article on page 25.)

H. D. BAYLOR, President, Louisville Cement Corp., Speed, Ind. (August 25, 1950). Representative of Louisville Cement company membership since 1920, and representative of his company for many years on Committees C-1 on Cement, C-12 on Mortars for Unit Masonry, and E-8 on Nomenclature and Definitions. He had a leading part in organizing Committee C-12 and did much to stimulate its work.

BASIL DUNGLINSON, Chief Chemist, Chicago By-Products Corp., Chicago, Ill. (May 12, 1950). Member since 1937.

HERBERT HENDERSON, retired Vice-President, Engineering Dept., Gulf Oil Corp., Pittsburgh, Pa. (July 7, 1950). Representative of company membership 1931-1950.

W. G. HOOPER, Vice-President, Midwest Piping and Supply Co., Inc., St. Louis, Mo. (March 11, 1950). Representative of company membership since 1937.

WILLIAM MUESER, retired Civil Engineer, Long Island City, N. Y. (August 4, 1950). His affiliation in A.S.T.M. dating from 1898, Mr. Mueser received his Fifty

Year Membership Certificate in 1948. For many years associated with and later owner of the Concrete-Steel Engineering Co., New York City, Mr. Mueser subsequently was associated with the Federal Administration of Public Works, Staten Island, as Engineer-Inspector and Construction Engineer, and during the recent World War served in the Engineer Research Office of the War Department, New York City.

CHARLES EDWARD SKINNER, retired formerly Assistant Director of Engineering, 'Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa. (May 12, 1950). Member since 1903. (See article on page 25.)

JOHN C. SOUTHARD, Director of Research, Solar Aircraft Co., San Diego, Calif. (September 4, 1950). A member of A.S.T.M. since 1948, and a member of the Aviation Panel of the A.S.T.M.-A.S.M.E. Joint Committee on Effect of Temperature on the Properties of Metals, Dr. Southard was serving his first year on the N.A.C.A. subcommittee on Aircraft Structural Materials, and was actively associated with many other technical societies. Formerly affiliated with the Titanium Alloy Mfg. Co., Niagara Falls, N. Y., he was the author of many technical papers, and had contributed several chapters of text and illustrations to the recently published book in the Pitman Metallurgy Series, "Titanium in Steel."

Charles Edward Skinner
1865-1950

IN THE death of Charles Edward Skinner, member of the Society since 1903, affiliated with Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa., from 1890 to 1932 when he retired, the Society loses one who throughout his membership of 48 years was constantly interested in its work. His wide range of interests in Westinghouse, which included among other things responsibility for the organization and equipment of Chemical, Physical, and Process Laboratories, as well as the High-Tension Test Laboratory and a new Research Laboratory, were reflected in his wide range of interests in the Society. He was an officer of the Society in 1912, and it was very heartwarming to receive from Dr. Skinner, when he came from retirement during the recent World War to assist the Signal Corps, U. S. Army, a handwritten note indicating how valuable he considered the A.S.T.M. work to be in connection with his War endeavors. He was vitally interested in the early International Association for Testing Materials, was an officer of the American Engineering Standards Committee, the forerunner of the A.S.A., and was a Past-President of the American Institute of Electrical Engineers.

Francis Edward Bash
1883-1950

IN THE death of F. E. Bash, long a member of ASTM, the Society loses one of its very active committee members and officers, who over a period of many years contributed a great deal to the advancement of the Society's work in both standardization and research. Ill for only a short time, he died on September 14 at Morristown, N. J., Memorial Hospital.

Born in the state of Washington, Mr. Bash received his technical education at the University of Washington and University of Wisconsin, accepting his first industrial position with Leeds & Northrup Co., Philadelphia. In 1923 he went with the Electrical Alloy Co. and was with the Driver Harris Co. for some 23 years, becoming Assistant Vice-President in Charge of the Technical Department.

While his most intensive work in ASTM was with Committee B-4 on Electrical Heating, Resistance, and Related Alloys of which he had been secretary virtually from the organization of the group over 20 years ago and to the work of which he made notable contributions, he also served effectively on the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals, A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys, and the Joint Committee on Filler Metal.

In addition to his valuable work in ASTM, he was an active member of numerous other technical and scientific groups including the American Chemical Society and Institute of Radio Engineers.

To his wife, his three sons, and two daughters the Society extends its sincere sympathy and records its appreciation for his many contributions to its work and the cordial spirit with which that help was given.

LABORATORY SUPPLIES...

Catalogs and Literature and Notes on New or Improved Apparatus

Note—This information is based on literature and statements from apparatus manufacturers and laboratory supply houses.

Catalogues and Literature

Large Two Lens Quartz Spectrograph—A new bulletin describes and illustrates all of the principal features of this precision research instrument. Dispersion curves for the interchangeable quartz and glass optics are included together with specifications, optical diagram, and list of recommended accessories. Instrument has become noteworthy because of its resolution and its continuous wave-length setting whereby any portion of the spectrum can be brought to the center of the plate. 8 pages.

Bulletin 151-50, The Gaertner Scientific Corp., 1201 Wrightwood Ave., Chicago 14, Ill.

pH Electrode Catalog—The complete line of pH electrodes and assemblies for both industrial and laboratory applications is now presented for the first time in a new, 28-page catalog, "pH Electrodes, Assemblies, Parts and Accessories." It lists the advantages of each type of electrode, and by means of an illustrated, double-page spread, shows each assembly and all of its components in tabular form. Glass electrodes for industrial use are shown in detail, whether for use in plastic, Pyrex, or iron assemblies; glass electrodes for laboratory use are illustrated with their appropriate measuring instruments. Other L&N Laboratory electrodes—hydrogen, quinhydrone, oxidation-reduction—are listed with their reference electrodes.

Catalog EN-85, Leeds & Northrup Co., 4934 Stenton Ave., Philadelphia 44, Pa.

Laboratory Equipment—A new 16-page issue of "What's New for the Laboratory" has just been released by the Scientific Glass Apparatus Co., Inc., of Bloomfield, N. J.—No. 11 in the series. It illustrates and describes a number of new items including a quadrant culture plate, a flask with special cork-insulated top to prevent burns to hands, and a vacuum oven that's heated by radiation, a new microscope-lamp featuring optical control of light intensity, a new type mortar and pestle, a tubular filter made of fritted glass, and introduces an ASTM certified thermometer testing set. All told, 20 items are illustrated and described.

Scientific Glass Apparatus Co., Inc., Bloomfield, N. J.

Proving Rings—The publication of an illustrated, descriptive bulletin on direct reading proving rings which are used in calibrating the loads of various types of testing machines and presses—including Brinnell and Universal testing machines is announced. This file-size data sheet gives full information on sizes, capacities, weights, and dimensions of these instruments. 2 pages.

Steel City Testing Machines, Inc., 8843 Livernois Ave., Detroit 4, Mich.

Instrument Notes

Metallograph—A recently announced metallograph is said to provide equipment that combines the economy of more limited capacity instruments with many of the performance advantages of the most advanced metallographs. Among the features stated by the manufacturer are: straight-line observation, centralized controls, new fine adjustment mechanism, improved heavy duty mechanical stage, new elevating device, improved filter and heat-absorbing system, solid cast optical bed. The metallograph includes equipment for bright field, dark field, and polarized light; metallographic phase contrast accessories can also be accommodated. Catalog E-232.

Bausch & Lomb Optical Co., Rochester 2, N. Y.

Ozonator for Tests of Rubber and Rubber-like Materials—Said to be the first precision ozonator ever to be made com-

mercially available, it has been developed through extensive research to meet a need for accurate ozone testing involving accelerated aging and weathering tests, cracking phenomena, etc. Among the design features are emphasized a uniform ozone and temperature distribution throughout the test space, a continuously operating electric flowmeter, and an accurate voltage control of the ozone generator. The air flow and the generator voltage are indicated by two meters mounted on the panel for convenience in maintaining constant test conditions.

G. F. Bush Associates, Box 175, Princeton, N. J.

Oscillograph—A quantitative, 10-megacycle cathode-ray oscillograph has been announced. Time calibration is provided for the horizontal sweep of the instrument and regulated voltage calibration for vertical deflection. Both time and voltage calibration are accomplished by substituting a calibrating signal for the input signal. The Y axis of the instrument, which includes a fixed, signal-delay line, provides a sensitivity of 0.1 volt peak-to-peak per inch. Transient response of the Y axis is such that a pulse having a rise time of 0.01 microsecond or faster will be reproduced with a rise time not exceeding 0.03 microsecond. The Y axis amplifier provides 2.5 in. of undistorted deflection of unidirectional pulses. An equivalent 5 in. is provided for symmetrical signals and may be positioned over the useful area of the cathode-ray tube. For either the time-base signal or signals applied externally, the direct-coupled X axis amplifier is said to provide undistorted deflection which exceeds the useful area of the screen.

Allan B. Du Mont Labs., Inc., 1000 Main Avenue, Clifton, N. J.

Automatic Colorimeter for Cotton—This instrument is built to show automatically, and without manipulation by the operator, the exact color of each sample of cotton brought to it. For cotton colors, coordinates "R_d" and "b" provide, with conversion, a picture close to that of measurements in terms of Munsell value and chroma. The instrument was designed to show graphically on a two-dimensional scale simultaneous values for reflectance and yellowness. The instrument is self-contained in a movable cabinet about table height, with a minimum of exposed parts. The exposure of samples and reading of results is done in the horizontal plane of the table. While this particular instrument is limited to the range of cotton colors, the principles upon which it is designed are adaptable to other limited ranges of color, in either two or three dimensions.

Henry A. Gardner Lab., Inc., 4723 Elm Street, Bethesda 14, Md.

Color Densitometer—A new color densitometer, a precision photometer with a wide range of diversified uses in many industries, has been announced. This instrument permits accurate measurement of color densities in narrow spectral ranges, as well as visual densities over an unusually wide range. With accessory equipment, reflection densities of opaque samples and transmission densities of liquids can also be measured. Applications for the color densitometer are said to be found in photography, chemistry, research, and industry.

Bulletin No. 144, The Macbeth Corp., Newburgh, N. Y.

Test Sieve Shakers—A new line of vibrating sieve shakers, using standard screen scale 8-in. testing sieves, according to the manufacturer, provides sieve analyses quickly and accurately, contributing to profitable operation, insuring customer satisfaction and a definitely uniform end product. They are quiet in operation, small in size, portable, and operate on ordinary 110-volt, a.c. (220-volt to order). The vibrating action of the shaker is produced by the patented electromagnet drive energized by rectified, half-wave a.-c.—3600 vibrations per min. from 60 cycle. There are no bearings, gears, belts, or pulleys, etc., to maintain and replace—no lubrication required. The capacity of the shaker is 6 standard 8- in. test sieves and bottom pan—or twice as many half sieves and bottom pan.

Syntron Co., Box 220, Homer City, Pa.

Recording Thermometer—A continuous record of actual temperatures in refrigerated trucks, railway cars, warehouses, ships, and retail stores' units, is said to be provided by a self-contained recording thermometer developed by the Tagliabue Instruments Division of the Weston Electrical Instrument Corporation.

The new instrument called the "Minicorder" is a completely self-contained recording thermometer with a temperature range, in various models, within the limits of -30 F. to $+165$ F. Charts are available for 24-hr., 72-hr., 3-day, and 7-day recording. Special two-pen models are made for on-off recording of related equipment. The instrument measures $5\frac{3}{4}$ by $5\frac{3}{4}$ by $4\frac{1}{2}$ in., and weighs $3\frac{1}{2}$ lb. Remote recording types are also available.

Tagliabue Instruments Div., Box 414, Weston Electrical Instrument Corp., Newark 5, N. J.

Radiation Pyrometer—A modified version of the conventional Brown Radiamatic pyrometer head to be used in conjunction with standard Brown "Electronik" potentiometers is designed to facilitate extremely high-temperature measurements, of the order of 3200 to 7000 F. The new unit is designed for sighting on relatively small targets (as small as $\frac{1}{8}$ in. in diameter) through a clear path or aperture of $\frac{1}{4}$ in. diam. This permits the unit to be used where measurements must be made inside of furnaces or other equipment with very thick insulation, and where the use of a large sighting hole cannot be tolerated because of excessive heat leakage.

Minneapolis-Honeywell Regulator Co., Industrial Div., Philadelphia 44, Pa.

Determination of Carbon-By-Combustion—Announcement has been made of the availability of a new electronic instrument for rapid and accurate determination of carbon by combustion. The instrument, called the "Combustron," is a compact, bench-mounted self-contained instrument; it comes fully equipped, ready to plug into the power supply. This new tool, according to the manufacturer, was developed and designed with the cooperation of leading steel chemists and metallurgists. Some of the features are: instant heating, rapid analysis, visible combustion, a sturdy Vycor reaction tube, and availability in one or two tube models. It operates on 115 or 230 volts—60 cycle, single phase, power supply. Burrell Bulletin No. 319 describes "Combustron."

Burrell Corp., 1942 Fifth Ave., Pittsburgh, Pa.

INSTRUMENT COMPANY NEWS . . . .

Announcements, changes in personnel, new plants and locations, and other notes of interest

BAUSCH & Lomb Optical Co., 635 St. Paul St., Rochester, N. Y., announces the promotion of three scientists: Harry G. Ott, head of the Military Engineering Dept. for the last two years, was appointed assistant to Carl L. Bausch, vice-president in charge of the company's Research and Engineering Div. and will be in charge of special military projects; Leon V. Foster (see personals); James R. Benford, a member of Bausch & Lomb's Scientific Bureau since 1935, was named as Foster's successor.

BOONTON RADIO CORPORATION, Boonton, N. J., announces the following changes in management, effective September 1, 1950; W. D. Loughlin has been elected Chairman of the Board of Directors, Dr. G. A. Downsbrough, President, and Dr. D. M. Hill, Vice-President in Charge of Research and Development.

SCIENTIFIC GLASS APPARATUS Co, INC., announces the transfer of its offices and plant to 100 Lakewood Terrace, about 1000 yards from its former address in Bloomfield, N. J. During the more than thirty years since the founding of the company, the old quarters had been enlarged three times, the last in 1941 when the plant was doubled in size. The increasing volume of business soon made those facilities inadequate. The new headquarters is a massive brick and concrete structure purchased in 1949. The floor area is over 100,000 square feet on one level, with a basement of equal size for storage. In converting the new plant, the layout has been planned for efficiency, with the flow of material moving progressively and in order. Production is carried out at one end of the building, while at the opposite end complete stocks of chemicals, instruments, apparatus, and glassware are stored in numbered bins ready for shipment.

TAYLOR INSTRUMENT COMPANIES announces the appointment of William I. Caldwell, member of the Research Department since 1937, as Director of Research. He takes the place of Nathaniel B. Nichols who has accepted a position as Professor of Electrical Engineering at the University of Minnesota. Since going with Taylor, Caldwell's work has been concerned largely with Bourdon springs, and the introduction of a 1200 F. mercury tube system by the company was made possible largely through his work. Recently he has been devoting most of his time to automatic control theory.

Rotary Screen for Laboratory Separation of Shives from Mechanically Prepared Wood Pulp[*]

By H. W. Greider,[1] R. A. MacArthur,[1] and L. C. Gischig[1]

Synopsis

This paper describes a laboratory screen for separation of shives according to their lateral dimensions from mechanically prepared wood pulp. (A wood shive is defined as a fiber bundle having a minimum lateral dimension.) The use of this screen in conjunction with the Bauer-McNett classifier, which evaluates fiber length, affords substantially complete estimation of the dimensional characteristic of mechanical wood pulp.

THE screen described in this paper was developed as part of a cooperative investigation by the members of The Asphalt Roofing Industry Bureau on the effect of shives in Asplund defibrated wood upon the weatherability and over-all quality of prepared roofings containing Asplund fiber. When such roofings are exposed to weathering, fiber bundles migrate to the surface of the roofing and leave holes or pits which can accelerate failure of the roofing. Shives also cause checking and breaking of the roofing felt during saturating and coating operations in the manufacture of asphalt roofings.

Based upon several years of observation of a series of roofings containing various amounts of hardwood and softwood Asplund fiber, the Research Committee of the Asphalt Roofing Industry Bureau has *tentatively* defined a shive as "a fiber bundle having a lateral dimension in excess of 0.015 in." Pending the results of continued observation on the effect of further aging of the exposed roofing, the fiber bundles of lateral dimension smaller than 0.015 in. are considered not to be deleterious in the roofing felt; it is further considered that the length of the fiber bundle is not a factor in the weathering resistance of the roofing.

Materials Tested:

In order to evaluate the performance of the screen, heat-treated Asplund defibrated, mixed-hardwood (containing oak, cottonwood, poplar, and catalpa); softwood (predominantly New Jersey pine); and heat-treated mixed-hardwood were screened.

Description of Screen:

The details of the shive screen in its present form are shown in Figs. 1 to 4.

The screen consists of a circular drum which rotates with its lower edge immersed in a pan of water. Water is supplied to the interior of the drum through a hollow drive shaft. The drum is so designed that one end can be easily removed, for introducing and removing the stock. The pan is supported on a separate shelf as shown in Figs. 3 and 4. The position of the surface of the water in the pan is shown in Fig. 1.

The cylindrical surface of the drum is made of No. 24-gage stainless steel[2]

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[2] Cylinder stainless steel stock is supplied by Harrington and King Perforating Co., Chicago, Ill.

Fig. 1.—Details of Screen.

SIDE ELEVATION

DETAIL OF SCREEN

.0145" SLOT

TANK (SOLDERED CONSTRUCTION)
MATERIAL - 20 GA STAINLESS STEEL
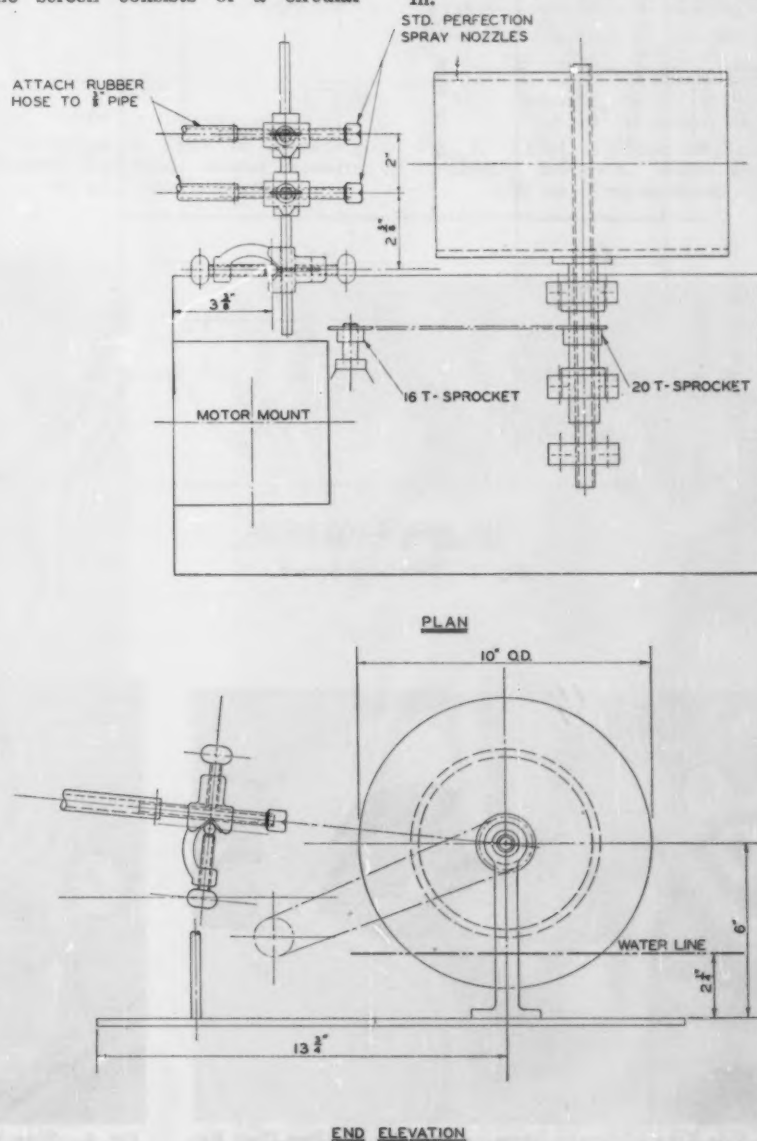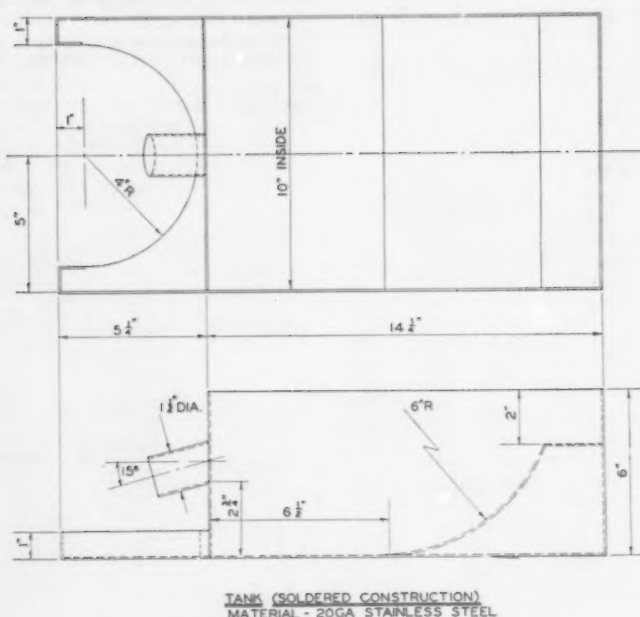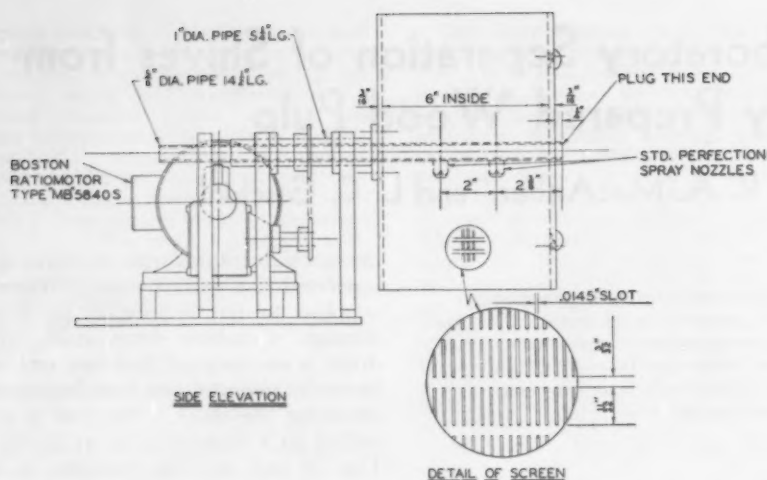
Fig. 2.—Details of Screen.

having parallel perforations measuring 0.0145 by $1\frac{1}{32}$ in., on $\frac{3}{64}$-in. centers, with the long dimension of the slots running circumferentially.

The drum is driven at 34 rpm. through sprockets by a Boston Ratiomotor[3] having a rated speed of 43 rpm.

Fibers and fiber bundles whose lateral dimensions are smaller than 0.0145 in. are washed through the perforated screen by water from the two internal Perfection No. 2 spray nozzles manufactured by Binks Manufacturing Co., Chicago, Ill. Each nozzle provides a spray angle of about 115 deg. and delivers about 28 gal. per hr. at a water pressure of 40 psi. The screen is kept clean by two externally mounted Binks Perfection No. 3 spray nozzles, each having a capacity of about 34 gal. per hr. at 40 psi. The overflow spout in the pan (see Figs. 3 and 4) is so placed as to hold the water level as shown in Fig. 1.

The material washed through the screen may be collected on a 325-mesh or similar size screen, for analysis in a Bauer-McNett classifier or for further examination of fiber length and diameter.

Procedure:

Present practice is to wash 10 g. (moisture-free basis) of prepared wood fiber in the screen for 45 min. with a water pressure at the spray nozzles of 60 psi. These conditions appear to provide a reasonably reproducible estimate of the shive content of the pulp. But it must be admitted that the determination is somewhat empirical, as is the case when evaluating fiber *length* in the Bauer-McNett classifier. The sample is prepared by taking a 10-g. representative sample of moisture-free
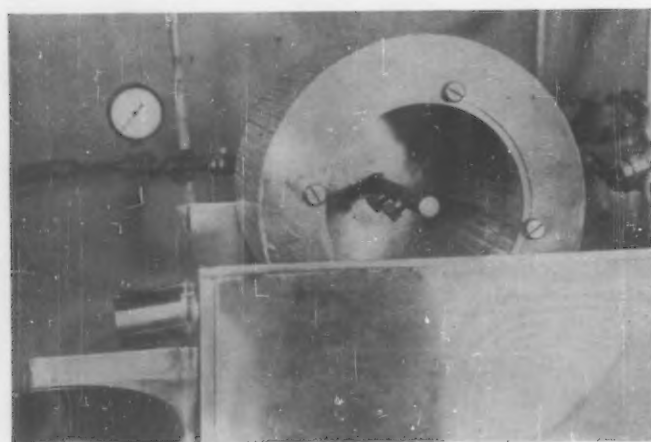
Fig. 3.—Side View of Screen Showing Showers and Pan Door Removed.
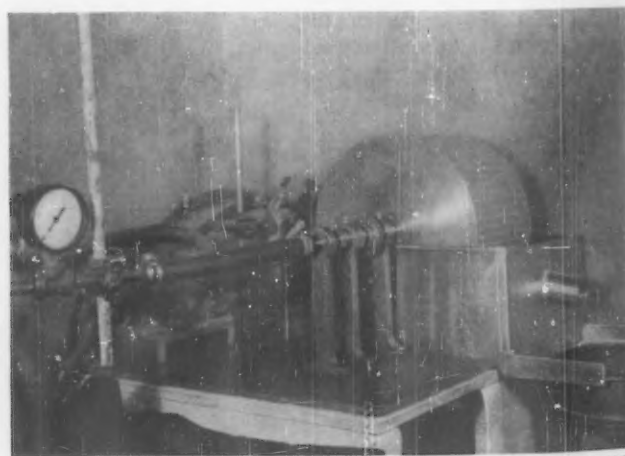
Fig. 4.—Side View of Screen Showing Hollow Drive Shaft with Water Feed Pipe.
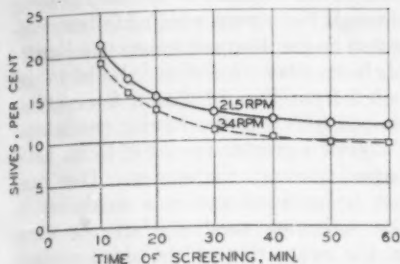
Fig. 5.—Effect of Screen Speed on Estimated Shive Content. Asplund Hardwood. Water Pressure, 60 psi.

stock which was prepared from a previously dried sample of defibrated wood weighing approximately 200 g. The 10-g. sample is mixed with 400 ml. of water, in a propeller type stirrer, until it is thoroughly dispersed. The mixing should not be so vigorous that the shives will be disintegrated.

After screening, the material retained in the screen is dried and weighed. This weight is considered to be the weight of shives present in 10 g. of the original stock.

Test Results:

The variables having effect upon the estimated shive content of a pulp include: (*a*) fiber sample weight,
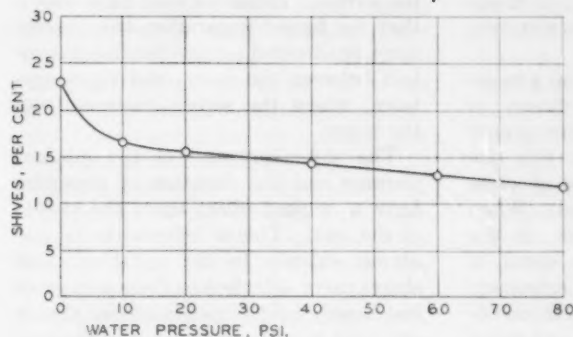
Fig. 7.—Effect of Water Pressure on Estimated Shive Content. Asplund Hardwood Pulp. Screened 45 min.
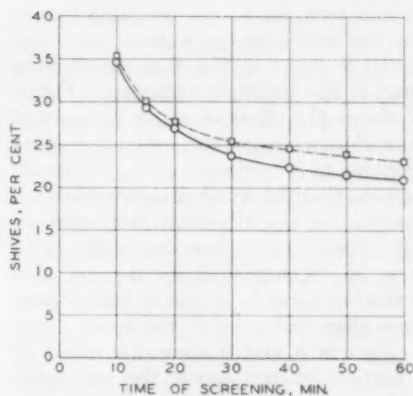
Fig. 8.—Effect of Time of Screening. Defibrated Hardwood. Water Pressure, 60 psi. (Two Samples).
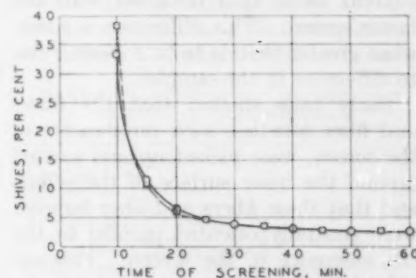
Fig. 9.—Effect of Time of Screening. Defibrated Softwood. Water Pressure, 60 psi. (Two Samples).

Fig. 6.—Asplund Hardwood Pulp Material Retained in Screen with 0.0145-in. Slots.
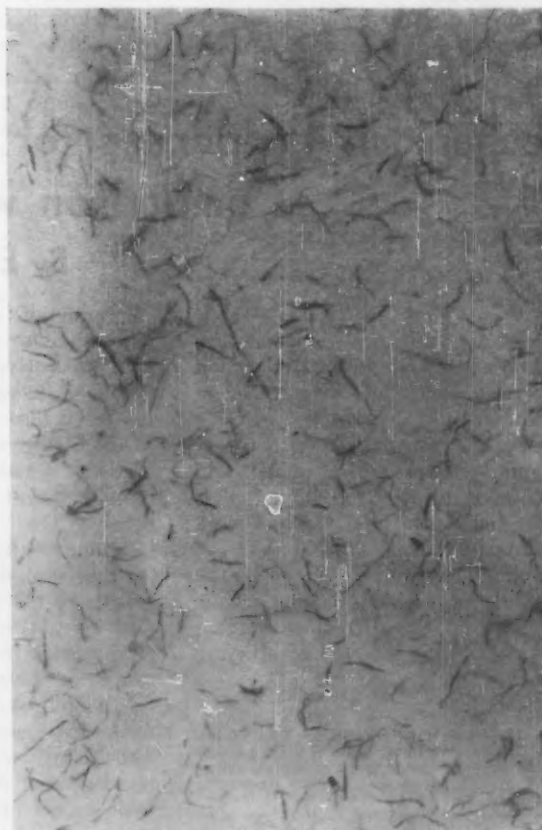
Fig. 10—Asplund Hardwood Pulp Material Washed Through 0.0145-in. Slotted Screen and Retained on 10-Mesh Bauer-McNett Screen.

(b) screen rotation rate, (c) internal spray direction, (d) nozzle pressure, (e) duration of the screening, and (f) orientation of perforations.

A weight of fiber of 10 g. (on a moisture-free basis) has been chosen as being of sufficient size to allow proper sampling of the pulp. Also this size of sample is not too large for effective screening in the time selected. When pressures greater than 20 psi. at the internal nozzles obtain, the speed of rotation of the screen is not extremely critical. Figure 5 shows the relation between the time of screening and speed of rotation on a hardwood Asplund pulp. It is to be noted that at the screening time of 45 min., there is a difference of about 2 per cent in the apparent shive content, the lower apparent content being that obtained with the faster screen. This difference is somewhat greater than is to be expected due to difference in the samples.

Early tests showed that the fibers and fiber bundles, with no showers in the screen, were picked up and carried around the inner surface of the screen and that these fibers and fiber bundles were generally oriented parallel to the circumference of the screen. Presumably because of this orientation, the screen with the long direction of the slot running circumferentially provides a faster separation of shives due to faster washing-through of the finer fibrous material.

The internal showers must obviously be directed in such manner as to force the smaller fibrous material through the screen. Extended tests have shown that for fastest separation this shower must be directed toward the line of contact between the screen and water surfaces, where the screen emerges from the water.

The water pressure of the internal showers and the duration of screening have a marked effect upon the results of the test. This is believed to be due almost entirely to the fact that most shives carry attached to them a more or less loosely held fibrous material that is removed by the action of the showers and which can pass through the screen if it is torn loose from the main bundle by the action of the water. Such material is shown in Fig. 6 as constituting part of the larger fiber bundles. Figure 7 shows the effect of water pressure at the showers upon the estimated shive content of a hardwood pulp. It would appear that the water pressure is critical and that a carefully controlled, arbitrarily selected pressure is necessary. This can be accomplished by the use of a reducing valve in a line of higher pressure than that used in the tests.

Figures 8 and 9 show the effect of duration of test upon the estimated shive content of hardwood and softwood Asplund pulps, respectively. These data are typical. From them, the screening time of 45 min. is indicated as adequate for relative estimation of shive content.

Figures 6 and 10 show, respectively, the shives separated from a typical Asplund defibrated pulp, and that portion of the material in the pulp passing through the screen which has been collected on the 10-mesh screen of a Bauer-McNett fiber classifier. It is to be noted from Fig. 10 that a few ribbon-shaped fiber bundles having one lateral dimension greater than 0.0145 in. have passed through the screen. This cannot be avoided and is a weakness in the screening method, but the error in the evaluation of the shive content of a pulp due to this factor is considered of little significance.

Experience with this type of separation indicates that more reproducible results could be obtained by running two screens in series, the first screen having an opening of about 0.030 in. in width. This screen would remove the larger shives which ordinarily have a great many loosely attached finer fibers that are torn loose in the present screen. Removal of these larger bundles before screening in the screen with the 0.0145-in. openings would greatly reduce the number of finer fibers produced in the screen by mechanical action.

Conclusions:

It would appear that a screen of the general type described will provide an adequate means of estimation of the amount of shives in mechanical wood pulp. Because of the somewhat empirical nature of the values obtained, all details of construction and use of the screen must be specified.

Mechanical Properties of Second-Growth Redwood and Comparison with Virgin Timber[1]

By Emanuel Fritz[2]

THE time is fast approaching when second-growth redwood timber, originating since logging began, will be cut and marketed. Already there are more than 100,000 acres of such young timber varying in age from under 50 yr. to nearly 100 yr. This does not include the larger area of more open stands composed almost solely of sprout-clump trees generated during the era of high-speed steam logging. Currently, with the use of tractors, logging is again of a type adapted to seedling

reproduction and consequently denser stands. During the recent World War a comparatively small volume of the older second-growth redwood was cut to meet the extraordinary war needs. This cutting ceased in 1947, fortunately, and the remaining stands are given an opportunity to increase their size and quality.

The eventual resumption of the cutting of second growth and the current efforts of the redwood lumber industry toward putting their operations on a continuous basis creates renewed interest in the properties of the wood of young redwood. This is the first of a proposed series of papers on second-growth redwood and on redwood forest management in general to make available an accumulation of material heretofore available only in typewritten or mimeographed form.

So-called second-growth redwood

(*Sequoia sempervirens*) is characteristically coarse grained due to its rapid growth. Since old-growth redwood, in contrast, is fine grained, the question is asked "How does the strength of the two compare?" The term "second-growth" redwood is a misnomer; nevertheless, it is well established and has important connotations. Old-growth timber is often referred to as "first growth" to distinguish it from "second growth."

Second growth is a term given to trees that arise following the logging of the old growth. Obviously, after such a violent change in the environment, each new tree starts out without competition and grows rapidly in diameter and height. In other regions similar results follow a great fire. If the new trees are widely spaced they should grow rapidly for several hundred years; if closely spaced, their initial rapid

growth gradually changes to a slower growth in response to the competition that develops as the trees become large enough for their roots to interlace and compete for soil moisture and their branch systems (crowns) interfere with one another to compete for light.

The term second growth is not exact. Lumbermen have extended its use to include not only coarse-grained trees originating after logging, but also those "original trees" that have grown rapidly from youth to 500 yr. or more and to the accelerated-growth portions of released trees. The term is therefore inaccurate and confusing. It would be better to use the terms "young timber" or "coarse-grained lumber."

As the existing old-growth timber nears depletion it will be replaced with young timber starting as seedlings or sprouts. Where the old-growth forest is selectively cut, marginal-size trees are left standing. These are from 100 to 500 yr. old and capable of accelerated growth rates. A new forest, following selective cutting, therefore will consist of young trees from sprouts and seedlings and of older residual trees from the original stand. That part of the growth of residual trees which follows selective cutting will be of coarser grain and will resemble the wood of real second-growth except that it will be free from knots. Its properties were not thoroughly investigated but they should parallel those of second growth of comparable growth rate and ring characteristics.

TEST TIMBER

The samples used in the strength tests, herein reported, were taken from a stand of 65-yr.-old second-growth redwood on Big River, Mendocino County, Calif. One hundred thirty trees were cut from a three-quarter acre plot (one tree was Douglas fir, a common associate of redwood, and three were residual redwoods from the original stand). The second-growth trees were of both sprout and seedling origin and varied in diameter, at breast height, from under 10 to 37.7 in. The heights of the dominant trees reached just over 150 ft. The stand was not fully stocked; nevertheless, on an acre basis, it yielded approximately 80,000 board feet of sawed lumber, dimension and cross ties; 47 per cent of the product was 1-in. lumber and 27 per cent was 2 in. thick. Since unmanaged young redwood, even in crowded stands, does not drop its dead branches promptly, it produces a lumber product predominantly of common grades.

TEST SAMPLES

The test samples were clear sticks taken from thick dimension material cut on the head saw and reduced to a 2 by 2-in. section, but being manufactured on a small jointer, their dimensions varied slightly. Exact dimensions were determined prior to placing the samples in the testing machine, and the exact cross-sectional area was used in computing the results.

The trees were cut and sawed in March, and the test samples were made shortly thereafter; however, it was not possible to make the tests until the following February, almost a year later, during which time the samples were permitted to dry out. All tests were therefore made on room-dry material with a moisture content, based on oven-dry weight, varying from 8.2 to 11.6 per cent, and averaging 9.5 per cent.

It is important to note that the trees producing the samples grew on an exceptionally good site, a deep river-bench silt. Such a site characteristically produces high yields per acre, and the product has the coarse grain generally associated with second growth. The grain is coarsest in the dominant trees and closest in the trees of the lower crown classes, that is, those subjected to crowding. Since the entire stand was of volunteer development and the dominant trees were deprived of full growing space, the outer growth rings were much narrower than those nearer the pith.

No record was made of the position of the samples in the log. The samples came from the butt logs of 8 trees and the second 16-ft. log of 2 other trees. Forty-six samples were tested in static bending (flexure); 34 in compression parallel to the grain; 10 in compression perpendicular to the grain; 42 in shear; and 44 for hardness. No impact or shrinkage tests were made. The hardness test was made in duplicate on the four sides and two ends.

PROCEDURE

All tests were conducted in the civil engineering laboratory of the University of California. Those for bending were made on a 30,000-lb. Olsen universal testing machine, using a deflectometer as pictured in U. S. Department of Agriculture *Bulletin 556*. A 50,000-lb. Olsen universal testing machine and a compressometer equipped with a dial indicator and made in the laboratory were used for the compression tests. Machine speeds were 0.02 in. per min. for static bending and 0.05 for shear. The dial for the compression tests read double the compression in 0.0001 in.

The test specimen dimensions, the methods of testing, and the methods of computing the results are those standardized by the A.S.T.M.[3] and

[3] Standard Methods of Testing Small Clear Specimens of Timber (D 143 – 49), 1949 Book of A.S.T.M. Standards, Part 4, p. 617.

the U. S. Forest Products Laboratory (2) and (4).[4] Since no strength tests were made on the wood in its green condition, the conversions of the strength values for the moisture condition at time of test to those at 12 per cent moisture content, the Forest Products Laboratory formula on p. 51 of *Bulletin 479* (2) and p. 61 of the Wood Handbook (4)

$$log\, S_D = log\, S_C + (C - D)\frac{log\,(S_B \div S_A)}{A - B}$$

was used, and the value for $log\,(S_B - S_A)$ and for A were taken from the same publication (2), pp. 54 and 51, respectively.

All strength data presented in the accompanying Table I are the averages for the several lots of test samples converted to a 12 per cent moisture content.

TABLE I.—STRENGTH OF SECOND-GROWTH REDWOOD.
(Based on 12 per cent moisture content)

Number of trees represented	10
Rings per inch	5.2
Summerwood, per cent	10.8
Moisture content, at test	9.5
Moisture content, green	127
Specific gravity, at test (0.35 at 12 per cent moisture content)	0.37
Specific gravity, based on green volume	0.34
Weight per cubic foot, at 12 per cent, lb.	25
Static bending (flexure):	
Stress at proportional limit, psi.	4 900
Modulus of rupture, psi.	7 700
Modulus of elasticity, psi.	1 250 000
Work to proportional limit, in.-lb. per cu. in.	1.34
Horizontal shear at maximum load, psi.	300
Compression parallel to grain:	
Stress at proportional limit, psi.	4 150
Maximum crushing strength, psi.	4 660
Compression perpendicular to grain:	
Stress at proportional limit, psi.	710
Hardness:	
End, lb.	530
Side, lb.	315
Shear, parallel to grain, maximum, psi.	1 090

MOISTURE CONTENT

As already noted, the samples were tested room-dry. The moisture content at test averaged 9.5 per cent, varying from 8.2 to 11.6 per cent based on the oven-dry weight, most of the samples having 9 to 10 per cent. All strength values were converted to a 12 per cent moisture content condition.

Moisture determinations were also made on the green lumber immediately after the logs were sawed. One hundred and fourteen samples from 19 trees were tested. Of these, 29 pieces were all sapwood and showed a moisture content of 197 per cent, the range being from 130.5 to 268.5 per cent; 63 pieces of all heartwood showed a moisture content of 145 per cent, the range being from 52.5 to 247.5 per cent; 22 pieces of mixed sapwood and heartwood had a moisture content of 126.8 per cent, the range being from 51.3 to 203.2 per

[4] The boldface numbers in parentheses refer to the list of references appended to this paper.

cent. For the entire lot the average moisture content for green wood is 155 per cent.

With respect to its green moisture content, second-growth redwood is comparable with old growth, whose moisture content is known to have an equally wide range and high average. During the milling experiment, at least 10 per cent of the logs proved to be sinkers, that is, so moisture laden as to sink in the pond.

Rings per Inch.—Since the samples were taken from the outer third of the logs, the rings are not as wide as they are nearer the pith. None of the samples was "fine grained" in the sense that old-growth redwood is fine grained, yet all old-growth redwood lumber is not fine grained (20 or more rings per inch); much of it has 8 or fewer rings per inch. The second growth here tested had from 2.25 to 12 rings per inch. Ten of the 46 samples had 4 or fewer rings per inch, and 12 had 6 rings or more per inch; 24 had between 4 and 6 rings per inch. The average for all 46 samples was 5.2 per inch.

Summerwood.—Of greater importance than ring width is the percentage of summerwood. It is characteristic of redwood in general to have a rather narrow band of summerwood. In this respect second growth differs little from old growth. The 46 samples showed summerwood to average 10.8 per cent, with a range from 5 to 21. Since the demarcation between springwood and summerwood is not always too closely defined and the summerwood band is so thin as to make measurement difficult, the percentages are indicative only; however, any error is against the summerwood percentage, measurements being made only where the change from springwood to summerwood was completed.

Specific Gravity and Weight.—Two figures are given for specific gravity—one for its volume at the time of the test and the other for its volume when green and at its maximum. For each case the moisture basis is the oven-dry condition. It varied from 0.27 to 0.41 for the green volume.

The conversion of the weight at test to that at 12 per cent moisture content was made under the assumption that "one-half per cent increase or decrease in weight accompanies an increase or decrease of 1 per cent in moisture content" (2).

The weight of the green wood, having an average moisture content of 127 per cent, is 48 lb. per cu. ft. The weight at 12 per cent is 25 lb.

ANALYSIS OF THE DATA AND COMPARISON BETWEEN OLD-GROWTH AND SECOND-GROWTH REDWOOD

Second Growth:

The second growth tested and herein reported was found to be slightly denser than that reported in *Bulletin 479* (no summerwood percentage is given for the latter) but considerably coarser in rate of growth. Stress in outer fiber at proportional limit and at maximum load, elastic resilience, maximum crushing strength, and hardness were found to be lower, while modulus of elasticity, stress at proportional limit for compression parallel to the grain, compression stress perpendicular to grain, and shear parallel to grain were higher than those reported for this type of wood in *Bulletin 479*. The ranges of the several strength values varied widely, as might be expected in such a variable material as wood; for example, the bending stress at proportional limit and the modulus of elasticity in bending ranged, respectively, from 3500 to 7780 psi., and 721,500 to 1,823,000 psi; the maximum crushing strength, parallel to grain, from 4600 to 7210 psi.; and the shear, parallel to grain, from 785 to 1640 psi. with close correlation between it and modulus of elasticity. This wide range of variability is further borne out by the ranges reported by Luxford and Markwardt in their *Bulletin 305* (1).

The number of trees producing the test pieces was 10 for the author's and 8 for the Forest Products Laboratory tests; however, the number of test pieces of the latter exceeded those of the author. All the author's trees came from the same acre of bottom-land, whereas the others came from widely separated areas and sites as well as from the entire length of the tree. No compression wood was involved in the present tests.

Old Growth:

Compared with wood from old-growth trees, the second-growth wood was found to be generally weaker except in one respect—shear—which is probably due to the tendency of young trees to be less straight grained than old-growth, except of course for those types of old-growth trees having interlocked grain. Second-growth properties compare with those of old-growth in the following ratios: specific gravity at 12 per cent moisture content, 0.35 to 0.40; weight, 25 to 28; bending stress at proportional limit, 4900 to 6900 psi.; bending stress at maximum load, 7700 to 10,000 psi.; modulus of elasticity, static bending, 1250 to 1340 psi.; elastic resilience 1.34 to 2.04 psi.; stress at proportional limit, compression parallel to grain, 4150 to 4560 lb.; maximum crushing strength, parallel to grain, 4660 to 6150 psi.; compression perpendicular to grain, 710 to 860 psi.; end hardness 530 to 790 psi.; side hardness, 315 to 480 psi.; and shear parallel to grain, 1090 to 940 psi.

Two pieces of old growth tested by the author as a check proved to be from 15 to 20 per cent under the old-growth values given above.

Second-growth redwood is not likely to be employed for as wide a range of uses as old growth. The lumber from unmanaged volunteer stands besides being coarse grained is quite knotty, and many of the knots are unsound. Its role will be largely for the less exacting purposes where great strength is not so important a factor. Where greater strength is required the intermingled Douglas fir is more desirable. However, under the intensive forest management that should obtain as present operations are placed on a permanent basis, selected second-growth trees (crop trees) can be held longer and so favored that they will produce a better quality of wood that should compare favorably, except in fineness of grain and therefore appearance, with the traditional old growth. It remains desirable, nevertheless, to encourage a larger per cent of Douglas fir in the future stands, so that the total product of the second-growth forest has a much wider range of utility.

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The Physical and Mechanical Properties of Second-Growth Douglas Fir[1]

By J. B. Alexander[2]

Synopsis

The increasing utilization of second-growth Douglas fir and the certainty that ultimately this type will supply practically the entire cut for the species brings into sharp focus the necessity for the determination of the physical and mechanical properties of such second growth. These trees are now established on an area approximating that of the remaining virgin stands in the Pacific Northwest—the province of British Columbia in Canada and the states of Washington and Oregon in the United States.

Data are presented for one shipment of seven logs, 60-yr. age class; one shipment of seven logs, 90-yr. age class; partial data for the species, virgin growth; partial data for one shipment of poles, 30-yr. age class; one shipment of poles, 60-yr. age class; and for one virgin-growth Douglas fir tree tested over its full commercial length of 152 ft. in 4-ft. sections.

Analysis of the test results shows that in the area sampled diameter growth is almost in direct proportion to age up to the 90-yr. age class; that growth rate is too rapid for high density and that density increases with increase in the radial distance from the pith.

For the area sampled, the average density is slightly less than the species average value for virgin-growth Douglas fir, although for the same rates of growth, that is, number of annual growth rings per inch, densities of second growth and virgin growth are almost identical.

There need be no concern regarding the adequacy of lumber cut from any section of second-growth Douglas fir for light construction. If the provisions of standard grading rules for structural timbers, which require a minimum rate of growth of 6 rings per inch, be strictly enforced, second-growth Douglas fir will possess adequate strength for heavy structural requirements.

STANDS of second-growth Douglas fir in the Pacific Northwest are assuming tremendous economic importance with the rapid depletion of virgin stands and their certain, ultimate extinction through the perfectly normal operation of harvesting the timber crop.

The total area of second-growth Douglas fir in British Columbia, Washington, and Oregon is estimated to be more than $6\frac{1}{4}$ million acres, an area roughly equivalent to that of the virgin stands. Harvesting of this material is under way already in many districts.

Regardless of the ultimate product— whether it be pulpwood, mine props, poles and piles, or sawlogs—it is essential to determine the physical and mechanical properties of the currently growing crop in order to put dependable data in the hands of all producers and consumers of wood and wood products. The silviculturist is as keenly interested

as is the manufacturer or the architect or engineer.

A minimum period of 30 to 40 yr. will be required to produce poles. At this age, in optimum growth conditions, Class 6 poles up to 45 ft. long can be expected. In the same conditions, trees in the 60-yr. age class will produce sawlogs 20 in., or more, in diameter. It is apparent, therefore, that it will require a period of 100 yr., or more, to produce sawlogs from which large structural timbers can be produced. On average to poor growth conditions, a much longer period will be required to produce trees with equivalent diameters.

Already it has been established definitely that Douglas fir trees 100 yr. old will produce little clear lumber—limbs persist too long. Although this is of considerable importance in many phases of the lumber industry, it is not of particular consequence in so far as structural design is concerned, for clear material is rarely used for heavy structural timbers.

The continuing existence and prosperity of the lumber industry, as well as the general prosperity of the entire Pacific Northwest, is dependent upon the continuing reproduction of new stands of various species, of which Douglas fir is the most desirable. Thus

it is vitally important to determine the physical and mechanical properties of such reproduction. It is equally important to know beyond doubt if the structure of this type of tree is compact and of great density, with correspondingly high mechanical strength, or if it is low in density (bulk without weight, low in mechanical strength and low in pulp content).

Scope

The data presented show results, based on standard tests of small clear specimens of virgin-growth Douglas fir and for second-growth Douglas fir of 60-yr. age class and 90-yr. age class, both from the same area. In addition, there are included partial data for two shipments of 25-ft. poles, one shipment coming from the same area as the second-growth logs, near Vancouver, British Columbia, the second from an area on Vancouver Island at a considerable distance.

As direct comparison with similar data for virgin growth is desirable, species average values are shown for Douglas fir in green and 12 per cent moisture conditions, as is partial data for the butt 12 ft. from one tree of virgin growth which has been tested for its complete commercial length of 152 ft.

For the 60-yr. age class logs, data are presented for green and for 12 per cent moisture conditions, whereas data for the 90-yr. age class cover the green condition only.

Description of Material

Seven logs were included in each of the shipments—60-yr. age group and 90-yr. age group. All were cut below 400 ft. altitude in the Fraser River Valley. Normal temperatures lie between $+15\,F.$ and $+90\,F.$ Infrequently a low of $+5\,F.$ or a high of 100 F. may occur. Rainfall is abundant on the area, averaging in excess of 50 in. annually. The area will rate Site Class II.

Trees were selected after a careful cruise of the area on which logging was in progress, and after examination of felled logs in the bush and those in log decks. The primary purpose in selection was to obtain a representative sample of material and a range in quality for the area sampled.

This practice in selecting logs differed somewhat from that laid down in

Fig. 1.—Typical Transverse Sections of Second-Growth and Virgin-Growth Douglas Fir.

From a shipment of virgin-growth Douglas fir grown on the same area, one tree was tested for its full commercial length of 152 ft. after cutting it into 4-ft. bolts. Bolts "A", "B," and "C," totaling 12 ft. in length directly above the stump, that have their mid-length at 6 ft. above the stump, provide a further direct comparison.

METHOD OF TEST

Individual test specimens were weighed and measured immediately prior to testing. They were tested in accordance with the provisions of A.S.T.M. Methods of Testing Small Clear Specimens of Timber.[3] Testing was carried out at room temperature, which varied but little from 70 F. Specific gravity and moisture content determinations were made for every test piece.

RESULTS

The various data are presented in the

TABLE I.—INDIVIDUAL TREE DATA.

Piece	Age, yr.	Diameter at Stump Inside Bark, in.	Stump Height, in.	Tree Height, ft.
60-YR. AGE CLASS				
No. 1..	60	20	12	134
No. 2..	58	22	12	140
No. 3..	58	21	12	138
No. 4..	61	22	12	142
No. 5..	58	20	16	133
No. 6..	62	18	15	140
No. 7..	62	21	18	147
90-YR. AGE CLASS[a]				
No. 1..	85	39	28	180
No. 2..	85	30	28	184
No. 3..	82	32	30	180
No. 4..	86	29	30	183
No. 5..	89	30	36	171
No. 6..	87	33	33	175
No. 7..	92	33	27	166

[a] One log of the 90-yr. age class showed abnormally low mechanical strength properties. Microscopic examination revealed innumerable compression failures and slip planes in the cell walls. This log was culled and no data from it have been used in computing averages.

the A.S.T.M. Standard Methods of Testing Small Clear Specimens of Timber (D 143 – 49).[3] One 12-ft. log was cut from each tree just above the stump. Each log was marked with the standard cruciform pattern on the end before it was sawed into carts which were cross-cut at their mid-length into two bolts marked "A" and "B", respectively, "A" being the butt section. Composite bolts were established by selecting test sticks from "A" and "B" bolts alternately. One such bolt was converted into standard test pieces which were tested in green condition, the other when at 12 per cent moisture content.

Butt logs were selected because the

[3] 1949 Book of A.S.T.M. Standards, Part 4, p. 617.
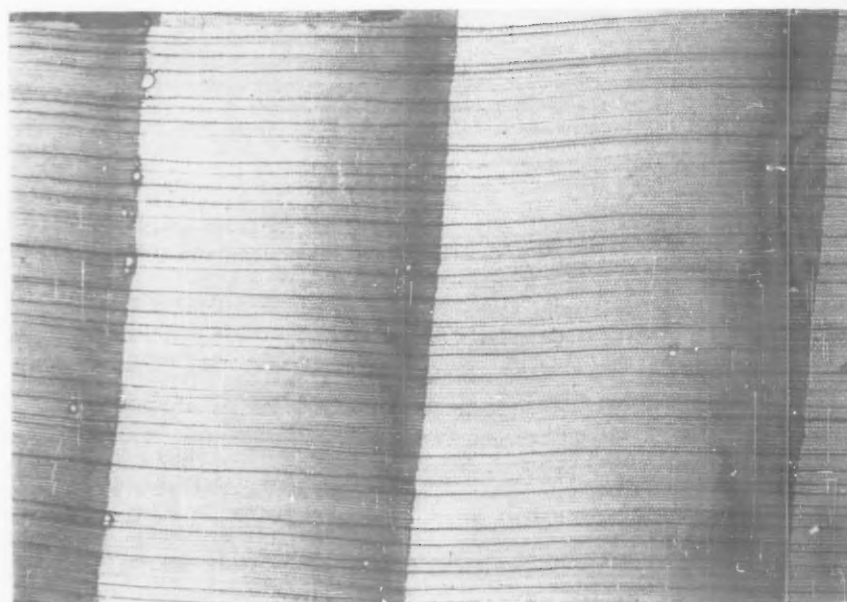
Fig. 2.—Transverse Section of Second-Growth Douglas Fir, 60-yr. Age Class ($\times$ 17.5). (Reduced to 70 per cent size in reproduction.)

25-ft. poles were loaded at the groundline, 5 ft. from their butt ends, and disks for the determination of specific gravities were cut just above this section so that the specific gravities were determined at a section approximating 6 ft. from the butt, thus approximating the mid-length section of a 12-ft. butt log. In addition, logs of the 60-yr. age class had been cut directly above the stump, because of their comparatively small diameter—20 in. average. Thus, no height factor has been introduced to raise doubts as to the propriety of direct and correct comparison of test data.

form of tables and figures as indicated below:

1. Individual tree data (Table I).
2. Tree and shipment averages for 60- and 90-yr. age classes; averages for bolts "A," "B," and "C," of tree 1, shipment 2, and species averages, virgin growth, all in green condition (Table II).
3. Tree and shipment averages, 60-yr. age class, and species averages, virgin growth, both at 12 per cent moisture content (Table III).
4. Rate of growth - specific gravity relations for 60- and 90-yr. age classes and for virgin growth (Table IV).

TABLE II.—TEST RESULTS, SECOND-GROWTH DOUGLAS FIR. PARTIAL DATA VIRGIN GROWTH.

Piece	Rings per inch	Proportion of Summerwood, per cent	Shrinkage Based on Green Measurements, per cent			Specific Gravity—Weight and Volume Oven-Dry	Moisture Content Based on Weight Oven-Dry, per cent	Static Bending			Impact Bending		Compression Parallel to Grain		Compression Perpendicular to Grain		Shear Parallel to Grain	
			Radial	Tangential	Volumetric			Number of Tests	Modulus of Rupture, psi	Modulus of Elasticity, psi	Fiber Stress at Proportional Limit, psi	Modulus of Elasticity, psi	Number of Tests	Maximum Crushing Strength, psi	Number of Tests	Stress at Proportional Limit, psi	Number of Tests	Maximum Stress, psi
Shipment 78—60-yr. Age Class, Green Condition																		
No. 1	6	36	5.4	8.3	11.9	0.498	56.2	11	7083	1 687 000	12 826	2 718 000	21	3621	6	531	6	817
No. 2	6	38	5.5	10.0	14.1	0.522	69.3	11	7578	1 715 000	11 290	2 804 000	22	3808	6	376	6	979
No. 3	5	36	5.5	11.2	12.2	0.483	46.9	11	7547	1 519 000	13 861	3 164 000	23	3571	5	408	6	961
No. 4	6	34	5.1	.....	12.3	0.480	44.0	10	7284	1 628 000	12 041	2 727 000	20	3665	6	463	6	904
No. 5	6	38	5.4	8.9	12.8	0.475	44.7	8	6455	1 498 000	10 241	2 446 000	17	3471	5	339	6	822
No. 6	6	39	5.0	10.5	12.6	0.456	46.4	7	6817	1 597 000	11 355	2 124 000	17	3427	6	417	6	773
No. 7	6	42	5.1	9.9	10.5	0.475	51.7	10	7246	1 383 000	12 953	2 617 000	20	3635	6	437	6	959
Avg.	6	38	5.3	9.9	12.3	0.484	51.7	..	7288	1 579 000	12 138	2 677 000	..	3609	..	426	..	886
Shipment 93—90-yr. Age Class, Green Condition																		
No. 1	5	39	4.2	6.6	10.6	0.459	50.6	17	6601	1 232 000	10 237	1 616 000	31	3270	14	385	6	833
No. 2	7	39	4.3	6.3	11.5	0.515	44.6	16	7999	1 625 000	10 824	2 047 000	27	3976	13	540	6	1122
No. 3	7	42	5.3	8.6	12.6	0.513	40.2	13	7386	1 597 000	11 145	2 044 000	30	3406	15	349	6	1003
No. 4	8	42	4.9	7.1	11.5	0.534	42.8	15	8355	1 924 000	11 894	2 248 000	31	4207	15	488	6	1062
No. 5	7	40	3.8	7.2	10.2	0.471	42.0	17	7437	1 457 000	11 810	2 097 000	30	3789	14	511	6	966
No. 6	6	46	4.6	7.0	11.8	0.526	45.0	17	7940	1 553 000	11 601	1 902 000	34	3752	16	453	6	1059
No. 7	7	34	3.6	5.5	11.8	0.472	38.2	20	7089	1 456 000	10 556	1 794 000	34	3368	16	431	..	875
Avg.	7	40	4.4	6.6	11.4	0.498	43.3	..	7520	1 538 000	11 149	1 964 000	..	3673	..	449	..	988
Species Average, Virgin Growth, Green Condition	17	39	5.0	8.1	12.1	0.52	41.0	..	7700	1 680 000	10 600	2 080 000	..	3760	..	510	12	910
Bolts A, B, and C (First 12 ft. Above Stump), Tree 1, Shipment 2—Green Condition	12	38	1.6	9.0	11.2	0.494	30.1	34	7701	1 635 000	8678	1 746 000	75	3852	20	627	12	924

TABLE III.—TEST RESULTS, SECOND-GROWTH DOUGLAS FIR.

Rings per inch	Proportion of Summerwood, per cent	Specific Gravity—Weight and Volume Oven-Dry	Moisture Content—Based on Weight Oven-dry, per cent	Static Bending			Impact Bending		Compression Parallel to Grain		Compression Perpendicular to Grain		Shear Parallel to Grain	
				Number of Tests	Modulus of Rupture, psi	Modulus of Elasticity, psi	Fiber Stress at Proportional Limit, psi	Modulus of Elasticity, psi	Number of Tests	Maximum Crushing Strength, psi	Number of Tests	Stress at Proportional Limit, psi	Number of Tests	Maximum Stress, psi
Shipment 78—60-yr. Age Class, 12 per cent Moisture Content														
6	27	0.481	11.3	9	14 148	2 261 000	16 033	2 788 000	22	7250	6	895	6	1372
6	29	0.504	11.9	11	12 770	2 080 000	11 589	2 081 000	22	7235	6	623	6	1700
5	26	0.471	11.8	11	11 782	1 794 000	14 880	2 472 000	23	6707	6	765	6	1433
5	28	0.469	11.6	10	11 881	1 999 000	14 996	2 459 000	19	6948	5	813	6	1229
6	32	0.463	11.6	8	10 662	1 748 000	14 069	2 158 000	17	6758	5	610	6	1302
6	28	0.455	11.8	7	11 900	2 043 000	15 652	2 564 000	18	6456	6	748	6	1231
6	28	0.463	11.6	9	11 909	1 890 000	13 743	2 489 000	21	6099	6	722	6	1556
Avg.	28	0.472	11.6	..	12 185	1 973 000	14 701	2 497 000	..	6789	..	741	..	1399
Species Average, Virgin Growth, 12 per cent Moisture Content	..	0.51	12.0	..	13 000	2 030 000	15 800	3 360 000	..	7470	..	920	..	1430
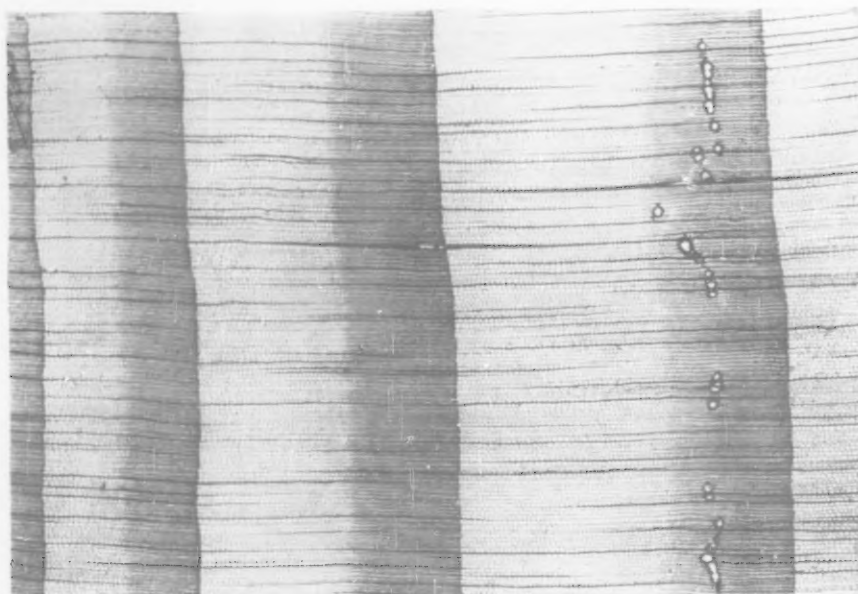
Fig. 3.—Transverse Section of Second-Growth Douglas Fir, 90-yr. Age Class (× 17.5). (Reduced to 70 per cent size in reproduction.)

TABLE IV.—RATE OF GROWTH-SPECIFIC GRAVITY RELATIONS.

Rings per inch...	3	4	5	6	7	8	9	10
60-YR. AGE CLASS								
Number of pieces...	8	29	34	32	21	9	5	4
Specific gravity[a]...	0.357	0.381	0.413	0.441	0.448	0.417	0.468	0.451
90-YR. AGE CLASS								
Number of pieces...	20	30	44	24	15	13	10	13
Specific gravity...	0.373	0.387	0.427	0.456	0.453	0.468	0.477	0.479
VIRGIN GROWTH								
Number of pieces...	3	6	10	10	17	15	27	29
Specific gravity...	0.431	0.440	0.447	0.466	0.479	0.472	0.495	0.498

[a] All specific gravities based on weight and volume oven-dry.

5. Age, diameter, and specific gravity relations for 60- and 90-yr. age classes, bolts "A," "B," and "C," tree 1, shipment 2, and for 30 and 60-yr. age classes of poles (Table V).

6. Variation in specific gravity, modulus of rupture, and maximum crushing strength with increasing radial distance from the pith (Table VI).

7. Typical transverse sections of second-growth and virgin-growth Douglas fir (Fig. 1).

8. Transverse sections of second-growth Douglas fir in 60- and 90-yr. age classes, magnification × 17.5 (Figs. 2 and 3).

9. Comparison of appearance for transverse sections of 90-yr. rapid and extremely rapid second-growth Douglas fir with those of virgin-growth Douglas fir (Figs. 4, 5, and 6).

10. Correlation of specific gravity and distance from pith tree center in second-growth Douglas Fir (Fig. 7).

11. Radial section of second-growth Douglas fir showing numerous compression failures in the cell wall, × 200 (Fig. 8).

ANALYSIS OF RESULTS

Standard test procedure was used throughout; thus the results obtained can be compared directly for all tests made on material in green condition and at 12 per cent moisture content, respectively. The slight difference in average moisture content in the latter case is insufficient to warrant any attempt to adjust data to 12 per cent moisture.

There is a remarkable agreement in the average specific gravities determined for 30-yr. age class, 60-yr. age class, 90-yr. age class, and for bolts "A," "B," and "C" of tree 1, shipment 2, virgin growth, 180-yr. age class. There is also a remarkable relation in diameter growth for the three age classes, the ratio being approximately 1 : 2 : 3 for age class and diameter.

For the second-growth material of shipments 78 and 93, specific gravities and strength properties were almost exactly equivalent at equivalent distances from the pith of the tree and for test pieces which showed the same number of growth rings per inch in the range from 3 up to, and including, 10 rings per inch. Although the second-growth Douglas fir tested showed a large percentage of summerwood generally, the specific gravity is not so great as might have been anticipated. A comparison of the lumens or cell cavities (Figs. 6 and 8) will disclose the reason. The cavities or lumens in Fig. 6, second growth, are very much larger than those in Fig. 8, virgin growth, although the cell walls in both are closely similar in thickness; consequently, there is less wood substance per unit of volume in the second growth and a lesser specific gravity.
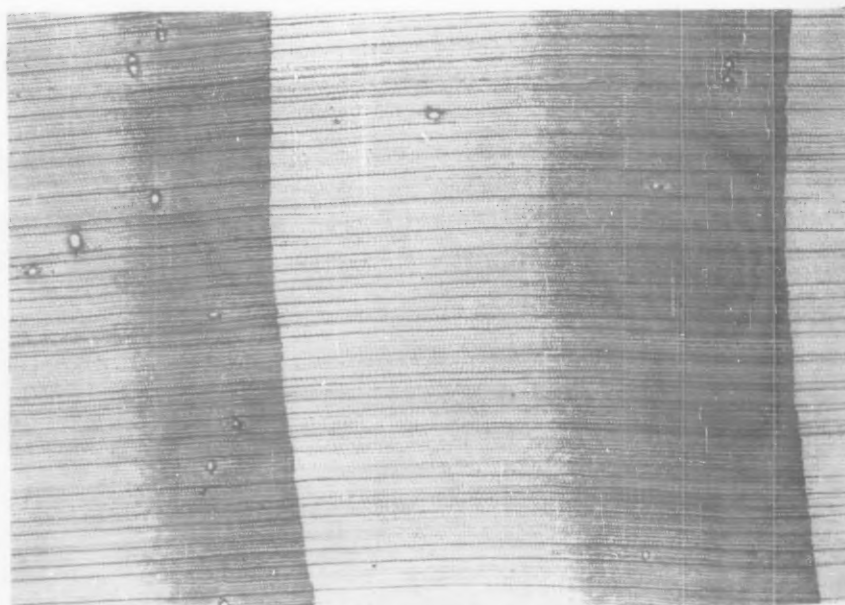
Fig. 4.—Transverse Section of Rapid Growth Second-Growth Douglas Fir, 90-Yr. Age Class (× 17.5). (Reduced to 70 per cent size in reproduction.)

Fig. 5.—Transverse Section of Extremely Rapid Growth of Second-Growth Douglas Fir, 90-yr. Age Class (× 17.5). (Reduced to 70 per cent size in reproduction.)

Material in Green Condition:

Modulus of rupture average for shipment 78 is 5.4 per cent less than the average for virgin growth; for shipment 93 it is 2.3 per cent less.

Fiber stress at proportional limit in impact bending is greater for both shipments of second growth than for virgin growth. The average maximum crushing strength in compression parallel to the grain was 4.1 per cent less for shipment 78 and 2.3 per cent less for shipment 93 than the average for virgin growth.

The greatest variation in strength properties is to be found in stress at proportional limit, in compression perpendicular to the grain, in which property the average values for shipments 78 and 93 were 16 per cent less and 12 per cent less, respectively, than the average value for virgin growth.

In shear parallel to the grain, the average unit values for shipment 78 and shipment 93 were 2.6 per cent less and 8.6 per cent greater, respectively, than the average for virgin growth.

There is significant increase in specific gravity with increase in the number of annual growth rings per inch, as shown by the values for 30-yr. age class and 60-yr. age class poles of approximately the same average diameter at load point, the 60-yr. group having 10 per cent greater specific gravity.

Air-dry (12 per cent Moisture) Condition:

Data for second-growth Douglas fir from shipment 78 only are available as yet. The average modulus of rupture in static bending for equipment 78 is 6.3 per

cent less than the average for virgin growth; for fiber stress at proportional limit in impact bending, 7 per cent less; for maximum crushing strength in com-

pression parallel to the grain, 9.1 per cent less, and for shear parallel to the grain, 2.2 per cent less. If the range in all strength properties within a species is taken into account, these percentage variations are not excessive.

Conclusions

The results presented in this paper must be considered to be of the nature of a progress report. They do not supply the complete answer with respect to the physical and mechanical properties of second-growth Douglas fir, as one area only has been sampled; nor will it be safe to assume that second-growth trees of the same age classes from other areas will bear to one another the same relative relationships in either or both physical and mechanical properties.

The following conclusions can be drawn from the results of this study. For the area sampled, the rate of growth for second-growth Douglas fir is practically constant up to the 90-yr. age class. Within the same limit, the average specific gravity is practically constant and is less than the average value for virgin growth. The high percentage of summerwood is not neces-

TABLE V.—AGE, DIAMETER, SPECIFIC GRAVITY RELATIONS, DOUGLAS FIR.

	Number of Pieces	Average Age, yr.	Average Diameter, in.	Average Specific Gravity[a]
60-yr. age class	7	60	21	0.484
90-yr. age class	7	87	31	0.498
Bolts "A," "B," and "C," tree 1, shipment 2	..	170	31	0.495
Species avg., virgin growth				0.52
25-ft. poles, 30-yr. age class	28	31	10	0.471
25-ft. poles, 60-yr. age class[b]	25	60	11	0.518

[a] Based on weight and volume oven-dry.
[b] This group from Vancouver Island.

Fig. 6.—Transverse Section of Virgin-Growth Douglas Fir Showing Sharp Contrast in Width of Annual Growth Ring, in Size of Cell Cavities, and Demarcation Between Springwood and Summerwood When Compared with Second-Growth (× 17.5). (Reduced to 70 per cent size in reproduction.)

Fig. 7.—Variation in Specific Gravity (Based on Weight and Volume Oven-Dry) with Distance from Pith of Tree.

sarily a true index of mechanical strength, as the summerwood cells have large lumens and consequently there are less of them per unit volume. Specific gravity increases outward from the pith. At equivalent distances radially from the pith, the average specific gravity for 60 and 90-yr. age classes is the same. For rates of growth between 3 and 10 annual rings per inch, both inclusive, the specific gravities, bending strength, and compressive strength are equivalent in the 60 and 90-yr. age classes for each growth rate. Except for growth rates of 3 and 4 rings per inch, the specific gravities within the same range for second-growth material are the equivalent of those for virgin growth. Specific gravity is influenced strongly by the rate of growth and increases with the increase in the number of growth rings per inch.

Although, for the area sampled, average values for the mechanical properties of second-growth Douglas fir are somewhat less than for the corresponding species averages for virgin-growth material, there need be no concern with respect to the adequacy of lumber cut from second-growth logs for light construction. If the standard provisions of structural-grading rules be strictly enforced, the requirement for a minimum rate of growth of 6 rings per inch will ensure adequate strength for heavy construction.

Rapid growth, without corresponding high specific gravity, is not desirable from the standpoint of the structural engineer or the pulp manufacturer; consequently, silviculturists should endeavor to promote growth practices which will result in rate of growth of 6 or more rings per inch, thus assuring a supply of second-growth Douglas fir which will possess adequate mechanical strength for the most exacting heavy construction, together with high specific gravity for high pulp yield per unit of measurement.

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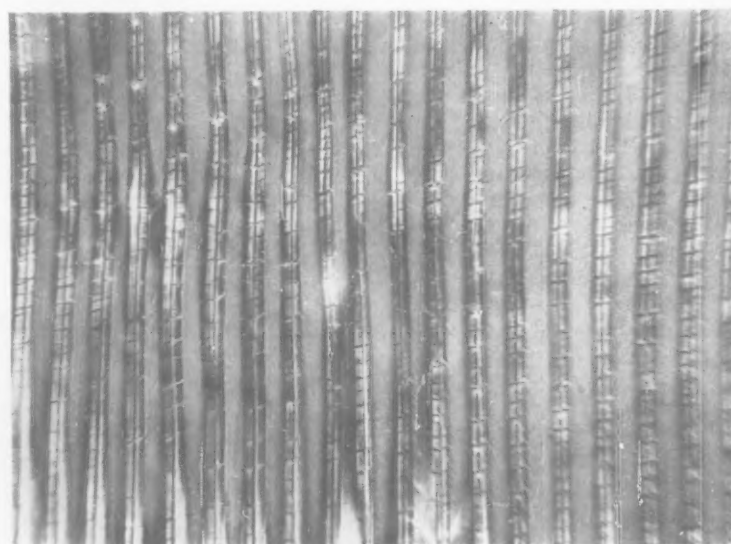
Fig. 8.—Radial Section of Second-Growth Douglas Fir Showing Numerous Compression Failures in the Cell Wall ($\times$ 200).

TABLE VI.—VARIATION IN PROPERTIES OF SAMPLE TAKEN AT INCREASING RADIAL DISTANCES FROM THE PITH.

Stick No.[b]	Number of Tests		Specific Gravity[a]		Modulus of Rupture, psi.		Maximum Crushing Strength, psi.	
	60-yr. Age Class	90-yr. Age Class	60-yr. Age Class	90-yr. Age Class	60-yr. Age Class	90-yr. Age Class	60-yr. Age Class	90-yr. Age Class
N1, N2; S1, S2............	27	25	0.379	0.380	6375	6268	3132	2839
N3, N4; E3, E4; S3, S4; W3, W4............	56	53	0.412	0.406	6931	6982	3415	3281
N5, N6; E5, E6; S5, S6; W5, W6............	46	56	0.447	0.441	7586	7629	3913	3792
N7, N8; E7, E8; S7, S8; W7, W8............	12	49	0.463	0.465	7961	8076	3950	4076
N9, N10; E9, E10; S9, S10; W9, W10.........	1	30	0.486	0.491	...	8088	4346	4138

[a] Based on weight and volume oven-dry.
[b] See D 143 – 49 for significance of stick numbers.

The Optimum Gypsum Content of Portland Cement[1]

Reported by Committee C-1 on Cement through its Working Committee on SO₃ Content

H. S. Meissner[2], Chairman

H. K. Cook
H. F. Gonnerman
W. C. Hansen

W. J. McCoy
Bailey Tremper
G. C. Wilsnack

IN 1946, William Lerch published the results of his studies on "The Influence of Gypsum on the Hydration and Properties of Portland Cement Paste."[3] His work indicated that there is an optimum gypsum content for each cement, and that, at the optimum, the cement will exhibit greatest strength, least shrinkage upon drying, and least expansion upon wetting. The optimum amount of SO₃ for each cement varies, being higher for cements of high-alkali content and for cements containing large amounts of calculated C₃A. High fineness was also shown to increase the optimum amount of SO₃ for cements of high, or moderately high C₃A content. As a result of Lerch's studies, both Federal Specifications and ASTM Standards for portland cement were amended to increase the maximum

properly regulated cement. As a result, Committee C-1 on Cement appointed a subcommittee to study the problem, and to present recommendations for action.

SCOPE OF STUDY WITHIN THE COMMITTEE

According to Mr. Lerch, the following criteria can be used to define the optimum quantity of gypsum in portland cement:

1. Early rate of hydration.
2. Strength.
3. Expansion in water.
4. Contraction during drying.

In addition, Mr. Lerch suggested a performance test to be applied to the finished cement, to show whether the cement actually contains the optimum amount of gypsum. The selection of a

Department of Highways, State of Washington, Olympia, Washington—Bailey Tremper.

Bureau of Reclamation, Denver, Colorado—H. S. Meissner.

Cements:

High- and low-alkali cements of each of types I, II, III, and IV, a total of eight cements in all, were used in the program. Type V was considered to be fairly well represented by the type IV cements. Each of these was prepared with seven different amounts of natural gypsum to provide SO₃ contents in 0.5 per cent increments from 1 to 4 per cent (except for type III cement, which contained 2 to 5 per cent). The cements were prepared by grinding clinker with low SO₃ content and also grinding a portion of the same clinker with gypsum to

TABLE I.—CHEMICAL ANALYSES OF CEMENTS.

Cement	Type	Oxide Analysis, per cent										Computed Compound Composition, per cent				
		SiO₂	Al₂O₃	Fe₂O₃	CaO	MgO	SO₃	Loss	Na₂O	K₂O	Mn₂O₃	CaSO₄	C₂S	C₃S	C₃A	C₄AF
No. 1	Type I, high-alkali	22.12	5.17	2.72	63.06	1.97	2.69	1.58	0.13	0.82	...	4.6	42.1	31.7	9.1	8.3
No. 2	Type I, low-alkali	20.98	6.08	2.13	62.65	2.81	2.49	2.01	0.15	0.12	0.58	4.2	44.4	26.7	12.5	6.5
No. 3	Type II, high-alkali	21.60	4.60	3.67	62.33	2.76	2.70	1.48	0.03	0.96	...	4.6	45.6	27.6	6.0	11.2
No. 4	Type II, low-alkali	23.59	3.74	3.33	62.78	1.78	2.62	1.87	0.20	0.30	...	4.4	39.1	38.1	4.3	10.1
No. 5	Type III, high-alkali	19.65	6.79	2.45	63.40	2.48	3.28	1.27	0.18	0.88	...	5.6	49.3	19.2	13.8	7.4
No. 6	Type III, low-alkali	19.45	5.73	3.37	62.58	2.52	3.44	2.26	0.14	0.11	0.45	5.8	53.7	15.3	9.5	10.3
No. 7	Type IV, high-alkali	24.79	3.89	3.03	62.45	0.75	2.66	1.30	0.91	0.39	...	4.5	27.7	50.2	5.2	9.2
No. 8	Type IV, low-alkali	26.34	3.18	2.74	62.23	1.78	2.03	1.41	0.12	0.30	...	3.4	21.8	59.2	3.8	8.3

SO₃ limit by 0.5 per cent when the cement contains more than 8 per cent calculated C₃A.

Lerch's work indicated that even these revised limits on SO₃ might be low, with the result that most manufacturers would be restricted in their efforts to secure the maximum benefit with respect to early strength and volume changes. In addition, his data implied that minimum limits on SO₃ content might also be in order, to assure the consumer of a

performance test would be based on the results of the criteria tests listed above, which are not entirely suitable for specification purposes. Obviously, such a performance test would have a distinct advantage over arbitrary chemical limits and would be easier to justify to both consumers and producers.

The following laboratories have participated in the study:

Concrete Research Division, U. S. Waterways Experiment Station, Clinton, Mississippi—H. K. Cook.

Portland Cement Association Chicago, Illinois—H. F. Gonnerman.

Universal Atlas Cement Company, Buffington, Indiana—W. C. Hansen.

Lehigh Portland Cement Company, Allentown, Pennsylvania—W. J. McCoy.

provide a high SO₃ content. These two cements were then blended to produce the various steps of SO₃ content. In some cases it was necessary to add gypsum to the cement to obtain the correct amount of SO₃. In these cases it was difficult to disperse the gypsum uniformly, and cements Nos. 3 and 7 were found later to contain large flakes of gypsum. No by-product gypsum was used in any of the tests. Cements Nos. 2 and 6 were ground in plant mills under normal production conditions. The remainder were ground in a laboratory ball mill. These cements were distributed to all participating laboratories.

All cements were ground to approximately 1900 sq. cm. per g. specific surface, except cements Nos. 5 and 6 (high early) which were prepared to a

TABLE II.—PHYSICAL

Cement	Type	SO₃, per cent	Autoclave Expansion, per cent	Wagner Surface, sq. cm. per g.	Air Content of Mortar C 185–47 Tᵃ	Compressive Strengths, psi. (Avg. for all Labs.)				Flexural Strength of Mortar Bars at 90 days, psi.
						1 day	3 days	7 days	28 days	
No. 1	Type I, high-alkali	1.06	+0.079	1910	10.0	...	1370	2520	4570	720
		1.50	+0.055	1900		...	1760	3040	4980	720
		2.23	+0.042	1895		...	2040	3140	4880	840
		2.69	+0.030	1890		...	2300	3310	4850	810
		3.21	+0.027	1895		...	2360	3380	4910	970
		3.55	+0.021	1880		...	2360	3410	4870	960
		3.97	+0.020	1875		...	2200	3220	4970	970
No. 2	Type I, low-alkali	0.98	+0.103	1785	5.4	...	1080	2150	3580	730
		1.48	+0.120	1760		...	1210	2440	4390	660
		1.96	+0.110	1735		...	1400	2440	4440	930
		2.49	+0.095	1745		...	1480	2540	4390	810
		2.99	+0.109	1725		...	1480	2460	4240	740
		3.46	+0.104	1745		...	1170	2360	4020	800
		3.97	+0.095	1770		...	830	1730	3870	750
No. 3	Type II, high-alkali	1.06	+0.087	1850	8.4	...	1670	2960	4860	780
		1.62	+0.083	1875		...	1830	3190	5230	890
		2.18	+0.070	1895		...	2330	3460	5240	1040
		2.70	+0.065	1920		...	2450	3600	5310	900
		3.06	+0.054	1930		...	2280	3350	4810	900
		3.62	+0.059	1945		...	1950	3030	4660	1030
		4.01	+0.089	1960		...	2490	3550	5110	950
No. 4	Type II, low-alkali	1.02	+0.018	1890	3.8	...	1340	2140	4620	890
		1.60	+0.009	1890		...	1490	2310	4720	770
		2.10	−0.011	1895		...	1680	2570	4670	990
		2.62	−0.019	1900		...	1570	2470	4510	990
		3.02	−0.018	1910		...	1300	2310	4360	1030
		3.63	−0.015	1915		...	1210	1950	4230	850
		3.97	−0.011	1920		...	1200	1750	3710	680
No. 5	Type III, high-alkali	1.92	+0.111	2085	8.0	1090	2790	4100	...	750
		2.42	+0.113	2090		1310	2890	4050	...	800
		2.77	+0.094	2095		1630	3130	4230	...	810
		3.28	+0.072	2100		1920	3430	4450	...	820
		3.79	+0.061	2105		1970	3500	4430	...	920
		4.32	+0.073	2110		1900	3580	4630	...	990
		4.90	+0.079	2110		1660	3710	4680	...	860
No. 6	Type III, low-alkali	2.10	+0.070	2600	9.2	1200	3010	4860	...	750
		2.46	+0.066	2600		1620	3200	4890	...	710
		2.91	+0.081	2645		1930	3240	4720	...	770
		3.44	+0.094	2700		1710	3260	4400	...	840
		3.92	+0.068	2650		1430	2830	4170	...	780
		4.40	+0.071	2600		1340	2230	3440	...	830
		4.94	+0.089	2510		1220	1950	2540	...	810
No. 7	Type IV, high-alkali	1.06	+0.020	1850	12.3	...	900	1460	3030	670
		1.45	+0.034	1875		...	1060	1950	3690	720
		1.94	+0.027	1900		...	1325	2170	3730	790
		2.66	+0.016	1925		...	1420	2310	3780	880
		3.11	+0.001	1950		...	1580	2300	3610	830
		3.39	−0.003	1915		...	1570	2340	3570	890
		3.93	−0.005	2000		...	1520	2310	3640	790
No. 8	Type IV, low-alkali	1.03	+0.018	1890	4.1	...	640	970	2590	850
		1.45	+0.005	1885		...	710	1000	2510	880
		1.72	−0.004	1885		...	820	1170	2570	850
		2.03	−0.005	1880		...	780	1270	2580	800
		2.82	−0.001	1880		...	590	1070	2760	800
		3.45	−0.003	1875		...	540	960	2650	750
		3.99	0.000	1875		...	540	920	2470	710

ᵃ 1947 Supplement to Book of A.S.T.M. Standards Part II, p. 229.

specific surface of about 2100 and 2600 sq. cm. per g., respectively.

TEST PROCEDURES AND RESULTS

Chemical Analyses:

The chemical analyses of the eight cements are shown in Table I.

General Physical Properties:

The results for the physical tests on the cements are shown in Table II, and are discussed briefly as follows:

Autoclave Expansion.—All cements were found to have exceptionally low autoclave expansions. It is interesting, though, to note that expansion in the autoclave was generally reduced as SO₃ content increased. This corroborates Mr. Lerch's finding that such reduction is greater than could be expected from a simple dilution of cement by gypsum.

Air Content of Mortar.—In types I, II, and IV, the high-alkali cements entrained considerably more air than did the companion low-alkali cements. No significance is attached to this difference in air content except as it affects durability in freezing-and-thawing tests.

Strength.—Compressive strengths were used as one of the criteria tests, and are discussed in more detail later. Tensile and flexural tests were made by one laboratory only. In general, the trends shown in the latter two tests were the same as shown by the compressive strength tests.

Normal Consistency.—The water requirements for normal consistency were found to increase slightly with increasing gypsum. Water requirements for plastic mortar showed the same effect.

Time-of-Set.—Both initial and final set were delayed as the gypsum was increased, except for cement No. 6 (type III, low-alkali).

Criteria Tests:

The purpose of the criteria tests was to establish the optimum gypsum content of each cement.

Early Heat Generation.—Two laboratories made tests for early heat generation, using the conduction calorimeter described by Lerch. The apparatus consists of a brass cup which is filled with freshly mixed cement paste. This cup rests upon a pedestal and is covered with a vacuum flask. The pedestal conducts the heat from the cup to a heat-dispersing water bath and, by means of resistance thermometers wound upon it, the heat generated in the paste is measured and recorded.

According to Mr. Lerch, a properly retarded cement, as indicated by early rate of heat liberation, will show two cycles of ascending and descending rates of heat generation which will not be appreciably changed by larger additions of gypsum. When three such

Average Initial Set, min.	Average Final Set, min.	Cumulative Heat of Hydration at 24 hr., cal. per g.		Average Expansion in Water, per cent			Average Contraction in Air, per cent			Extraction Tests SO₃, g. per liter				Cement
										Mortars		Neat Cements		
		Lab. No. 1	Lab. No. 2	7 days	28 days	90 days	7 days	28 days	90 days	18 hr.	24 hr.	18 hr.	24 hr.	
75	178	25.8	29.2	0.007	0.010	0.015	0.058	0.117	0.142	0.01	0.09	0.02	0.02	No. 1
148	262	32.3	35.8	0.007	0.010	0.015	0.054	0.114	0.141	0.00	0.01	0.02	0.02	
144	273	35.9	43.4	0.005	0.009	0.013	0.045	0.095	0.132	0.01	0.01	0.05	0.05	
169	286	43.3	48.5	0.005	0.008	0.010	0.037	0.083	0.110	0.00	0.01	0.23	0.14	
163	313	44.6	48.9	0.003	0.006	0.009	0.036	0.073	0.099	0.32	0.06	0.89	0.61	
183	321	42.4	45.7	0.008	0.010	0.013	0.036	0.073	0.099	1.48	0.68	2.50	1.96	
184	328	39.5	44.9	0.014	0.016	0.018	0.039	0.076	0.101	2.06	1.65	4.01	3.41	
7	168	29.5	24.2	0.010	0.018	0.023	0.062	0.122	0.149	0.01	0.00	0.01	0.01	No. 2
181	336	34.6	29.3	0.007	0.012	0.018	0.059	0.117	0.139	0.01	0.00	0.00	0.00	
198	334	39.1	38.2	0.006	0.011	0.016	0.051	0.104	0.126	0.04	0.01	0.00	0.01	
206	354	32.1	36.4	0.003	0.008	0.015	0.050	0.092	0.114	0.00	0.00	0.00	0.00	
197	361	29.2	32.2	0.010	0.015	0.019	0.049	0.087	0.108	0.52	0.20	0.43	0.13	
217	364	29.3	31.8	0.022	0.026	0.030	0.047	0.086	0.112	0.94	0.74	1.08	0.91	
225	374	28.8	29.3	0.046	0.050	0.052	0.056	0.095	0.119	1.02	1.05	1.21	1.22	
98	237	30.1	30.2	0.010	0.016	0.020	0.071	0.111	0.129	0.01	0.00	0.02	0.01	No. 3
200	325	32.9	37.6	0.006	0.010	0.015	0.058	0.096	0.113	0.01	0.00	0.02	0.01	
212	363	37.9	46.6	0.006	0.009	0.014	0.051	0.084	0.101	0.01	0.01	0.07	0.06	
222	370	40.4	48.3	0.005	0.009	0.013	0.044	0.073	0.092	0.19	0.00	0.44	0.26	
213	354	37.4	40.2	0.007	0.012	0.017	0.040	0.071	0.091	1.22	0.53	2.09	1.53	
243	374	36.4	41.0	0.014	0.017	0.021	0.048	0.079	0.099	1.65	1.15	3.00	2.46	
241	365	45.2	42.7	0.020	0.022	0.025	0.048	0.076	0.096	1.67	1.08	3.29	2.70	
246	402	28.0	28.8	0.007	0.010	0.014	0.050	0.087	0.106	0.00	0.00	0.01	0.00	No. 4
262	406	32.0	35.0	0.006	0.009	0.011	0.037	0.072	0.090	0.01	0.00	0.03	0.01	
262	401	31.5	34.8	0.006	0.008	0.010	0.033	0.067	0.084	0.25	0.05	0.36	0.15	
271	420	30.4	33.1	0.009	0.010	0.014	0.034	0.066	0.084	1.02	0.45	1.21	0.83	
274	419	29.8	33.5	0.012	0.014	0.018	0.045	0.076	0.092	1.07	1.05	1.27	1.33	
274	428	29.6	30.7	0.016	0.019	0.021	0.050	0.085	0.101	1.08	1.14	1.30	1.30	
284	414	30.3	32.4	0.017	0.022	0.024	0.056	0.092	0.110	1.13	1.19	1.29	1.34	
137	258	49.0	47.5	0.005	0.007	0.011	0.076	0.116	0.140	0.05	0.01	0.05	0.06	No. 5
140	279	51.8	50.4	0.006	0.007	0.010	0.074	0.118	0.145	0.02	0.01	0.05	0.08	
148	293	53.0	55.4	0.006	0.008	0.009	0.067	0.110	0.135	0.01	0.01	0.12	0.11	
144	286	56.1	60.2	0.006	0.006	0.008	0.061	0.104	0.127	0.01	0.01	0.31	0.13	
150	296	56.7	62.3	0.004	0.005	0.008	0.055	0.092	0.116	0.07	0.01	1.23	0.77	
136	295	55.6	57.0	0.006	0.007	0.010	0.051	0.086	0.105	0.92	0.10	3.08	1.54	
145	295	53.7	56.8	0.012	0.011	0.015	0.053	0.086	0.104	2.24	0.94	4.99	4.00	
100	222	43.2	43.0	0.008	0.011	0.014	0.083	0.120	0.136	0.01	0.01	0.01	0.01	No. 6
88	212	44.3	46.6	0.009	0.010	0.014	0.077	0.112	0.127	0.01	0.01	0.01	0.01	
81	204	49.5	51.9	0.008	0.011	0.014	0.066	0.102	0.119	0.01	0.01	0.03	0.02	
53	210	47.7	50.7	0.009	0.012	0.016	0.055	0.090	0.107	0.19	0.06	0.43	0.29	
39	232	44.5	48.4	0.013	0.016	0.020	0.057	0.089	0.105	0.91	0.55	1.44	1.18	
38	214	42.0	46.2	0.022	0.023	0.026	0.070	0.101	0.116	1.17	1.14	1.44	1.46	
54	229	39.8	43.4	0.026	0.038	0.042	0.076	0.120	0.140	1.18	1.19	1.47	1.49	
30	203	18.2	18.7	0.006	0.010	0.017	0.076	0.105	0.122	0.01	0.01	0.01	0.01	No. 7
176	317	27.9	23.9	0.006	0.009	0.015	0.067	0.106	0.122	0.01	0.01	0.01	0.01	
189	357	33.5	29.8	0.007	0.010	0.014	0.063	0.105	0.120	0.01	0.01	0.02	0.03	
219	409	38.8	38.7	0.005	0.006	0.010	0.050	0.092	0.110	0.02	0.01	0.12	0.13	
232	434	38.8	40.9	0.002	0.005	0.008	0.048	0.089	0.106	0.07	0.03	0.35	0.30	
227	428	38.8	40.4	0.002	0.004	0.007	0.043	0.083	0.098	0.93	0.10	1.40	0.89	
226	427	37.0	39.4	0.006	0.008	0.012	0.043	0.083	0.100	2.12	0.99	3.36	2.34	
284	423	21.6	24.3	0.006	0.010	0.015	0.052	0.087	0.099	0.02	0.01	0.01	0.01	No. 8
274	447	24.1	28.5	0.005	0.007	0.013	0.032	0.067	0.082	0.01	0.01	0.03	0.01	
280	425	24.8	27.4	0.004	0.007	0.012	0.035	0.068	0.084	0.34	0.15	0.37	0.16	
290	471	21.8	26.3	0.008	0.010	0.015	0.038	0.077	0.094	0.94	0.71	1.15	1.04	
303	474	21.4	25.5	0.015	0.017	0.021	0.054	0.088	0.108	1.09	1.09	1.23	1.26	
310	497	23.0	28.2	0.018	0.024	0.027	0.057	0.103	0.123	1.09	1.12	1.25	1.25	
310	489	22.9	26.8	0.022	0.031	0.036	0.058	0.110	0.129	1.11	1.13	1.25	1.30	

peak rates of heat liberation occur, it is generally with cements whose SO₃ content is insufficient. Additional gypsum added to such a cement will often reduce or eliminate the third peak. The first ascending and descending rate occurs within the first 5 or 10 min. after mixing the paste, and is extremely high but of limited duration. It is omitted from the plotted data which reflect only the second and following heat rates.

The heat liberation rate failed to indicate clearly the optimum gypsum content for any of the experimental cements except for cement No. 2. Therefore, the curves for cement No. 2 are the only ones included in this report. These are shown as Figs. 1 and 2. From these curves it is judged that the optimum for cement No. 2 was 3 per cent SO₃, according to one laboratory, and 2.5 per cent according to the other. For all cements, the 24-hr. cumulative heat of hydration was found to correlate quite well with other criteria, the highest heat occurring at the optimum SO₃ content. The cumulative heats at 24 hr. are shown in Table II. Since heat of hydration and strength are closely related at early ages, this provides an indication of the SO₃ content for highest 1-day strength.

Strength.—Plastic mortar cubes were made on all cements, according to the procedure described by ASTM Standard Method of Test for Compressive Strength of Hydraulic-Cement Mortars (C 109 – 47),[4] and tested at 3, 7, and 28 days for cements Nos. 1, 2, 3, and 4 (types I and II), at 1, 3, and 7 days for cements Nos. 5 and 6 (type III), and at 7 and 28 days for cements Nos. 7 and 8 (type IV). Three cubes were made by each laboratory for each age. The

[4] 1947 Supplement to Book of ASTM Standards, Part II, p. 43.

average strengths are shown in Table II. All cements show that highest strength is developed with some particular SO₃ content, although the results at different ages are not always in agreement. An illustrative plot of strength results is shown in Fig. 3. From inspection of all the data, optimum SO₃ values were selected for each cement and are included in Table III. In each case, the high-alkali cement requires from 0.5 to 1.0 per cent more SO₃ for highest strength than does its low-alkali counterpart.

One laboratory made flexural strength tests on 1 by 1 by 10-in. plastic mortar bars after they had been cured in water for 90 days. These results are listed in Table II. The amount of SO₃ in the cement yielding highest flexural strength is tabulated in Table III. In general, this optimum SO₃ content is close to that selected for highest compressive strength.
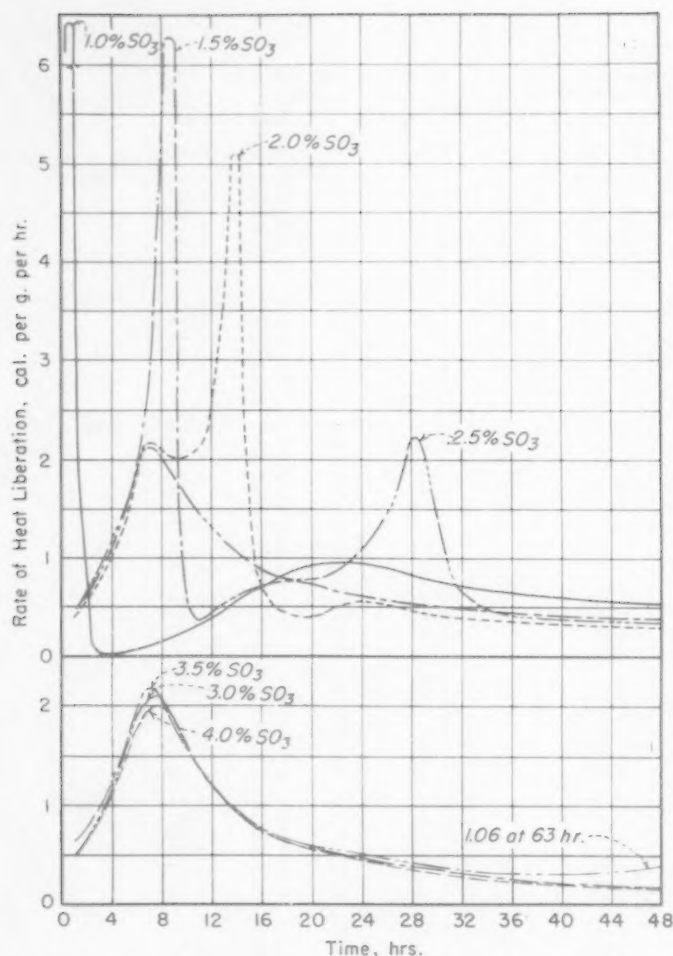
Fig. 1.—Rate of Hydration of Cement No. 2 by Laboratory No. 1.　　Fig. 2.—Rate of Hydration of Cement No. 2 by Laboratory No. 2.
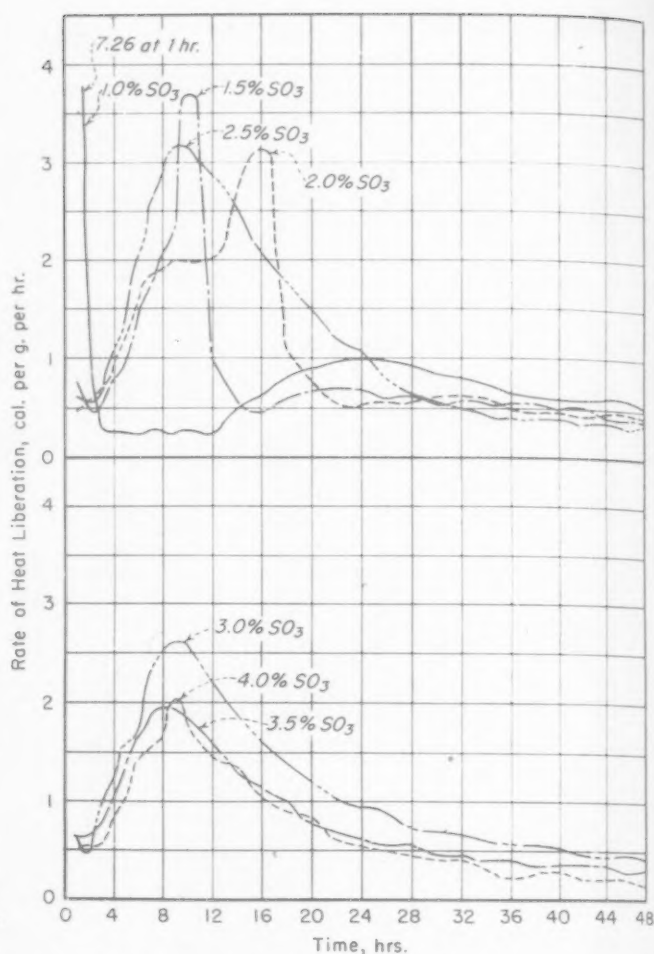
Expansion and Contraction Tests:

Plastic mortar prisms 1 by 1 by $11\frac{1}{2}$-in. (10-in. effective gage length) were prepared using the mix proportions $(1:2.75)$ and consistency required by ASTM Method C 109 – 47.[4] Four bars were fabricated from each mix. Two bars were used to measure the expansion of the mortar when stored in water, and the remaining two for contraction when stored in air. All specimens made for expansion measurements were cured in the mold at 100 per cent relative humidity and 70 F. for 1 day, then stripped, and measured for initial length. They were then stored continuously in water at 70 F. The bars used for contraction measurement were cured in the mold 1 day at 100 per cent relative humidity and 70 F., then stripped and placed in 70 F. water for 6 days, then removed from the water, given their initial reading, and stored continuously in air at 70 F. and 50 per cent relative humidity. Length measurements on these bars were taken at 7, 14, 21, 28, and 90 days from the time of their initial reading. Four additional specimens were made on a succeeding day to determine reproducibility of results. The averages for all laboratories are shown in Table II. Illustrative plots of the expansions and contractions are shown in Figs. 4 and 5. Distinct minimum expansion and contraction are to be observed for each cement, and the agreement between laboratories on the location of the minimum was very good. The optimum SO_3 contents for least expansion and contraction, selected by inspection of all the data, are shown in Table III.

It will be noted that large expansions due to wetting, and increased contractions due to drying may occur if gypsum is added in quantities in excess of the optimum. The indicated optimum quantities are, however, usually much greater than the maximum amount of SO_3 now permitted by specifications. If the optimum SO_3 content is used, cement of greater volume constancy should result, without risk of excessive volume change.

Extraction Tests:

The extraction tests were proposed by Mr. Lerch as possible specification performance tests to ascertain whether the cement contains optimum gypsum. They are not to be considered as one of the criteria tests, inasmuch as the time of extraction can be varied as required in order to make the results coincide with the results of the criteria tests. Extraction tests can be performed within 3 days, and do not require elaborate equipment, factors which make them preferable to the strength, expansion, contraction, and heat tests which have been described. Extraction tests were used also by W. C. Hansen and J. O. Hunt in studying "The Use of Natural Anhydrite in Portland Cement."[5]

In this study, both neat and mortar specimens were used. The neat cement mixtures were made according to ASTM Standard Method of Test for Normal Consistency of Hydraulic Cement (C 187 – 44).[6] Excess mortar from the fabrication of the length-change bars made according to ASTM. Method C 109 – 47[4] were used for the mortar mixes.

The extraction test is designed to detect the presence or absence of gypsum

[5] ASTM Bulletin, No. 161, October, 1949, p. 50.
[6] 1946 Book of A.S.T.M. Standards, Part II, p. 70.
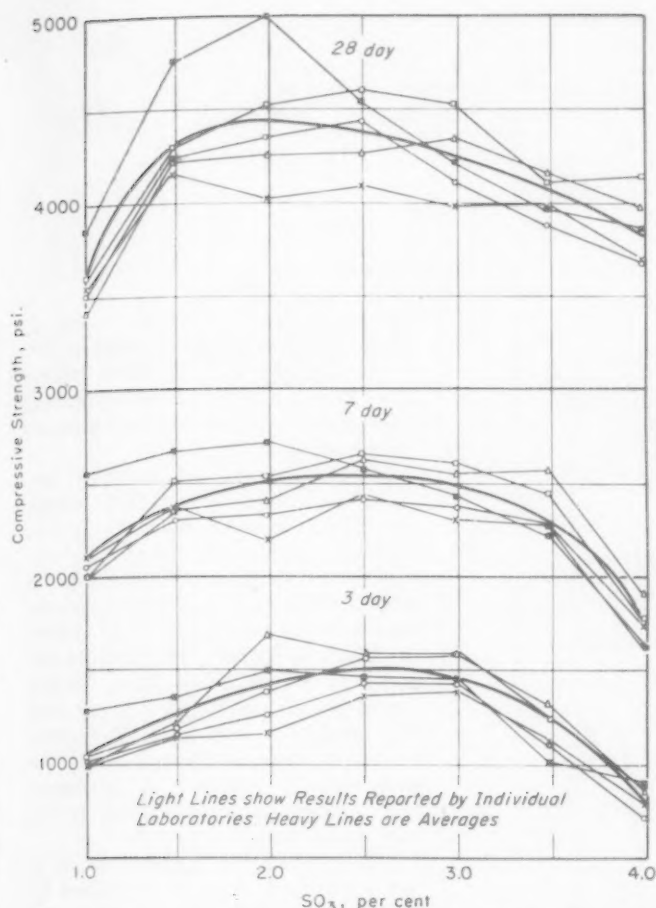
Fig. 3.—Plot of Compressive Strength Results of Cement No. 2.
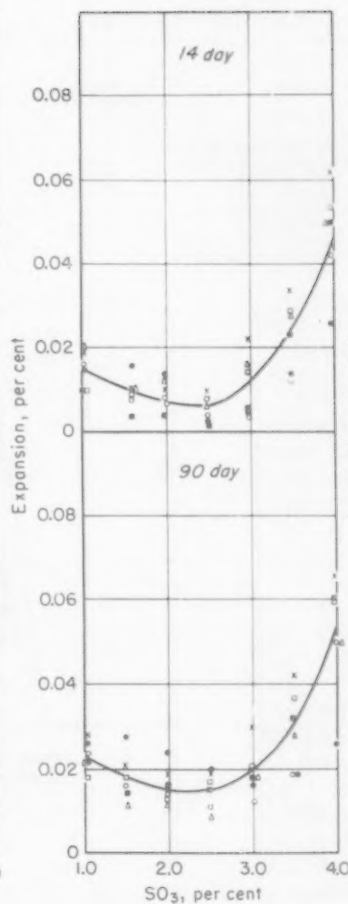
Fig. 4.—Expansion in Water of Cement No. 2.
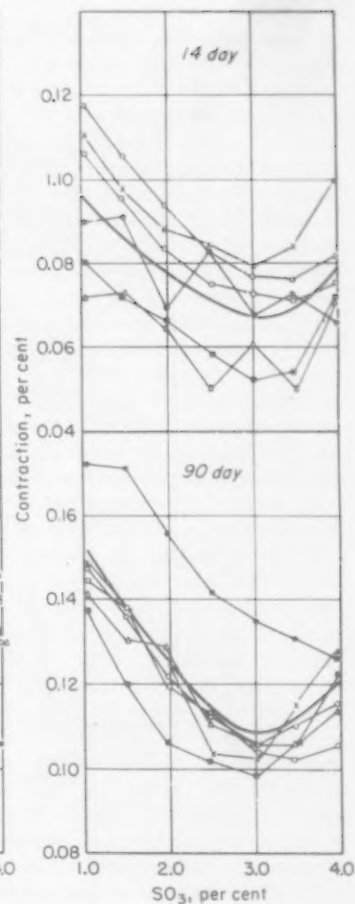
Fig. 5.—Contraction in Air of Cement No. 2.

in pastes or mortars that have been allowed to harden in the moist room at 70 F. for 18 ± ½ hr. and 24 ± ½ hr. The test is based on the relative SO₃ concentration of a solution saturated with respect to calcium hydroxide and calcium sulfate or sulfoaluminate. In the absence of alkalies, the SO₃ concentration of a solution saturated with respect to both gypsum and calcium hydroxide is approximately 1.0 g. per liter, whereas a solution saturated with calcium hydroxide and calcium sulfoaluminate, after the gypsum has become depleted, has an SO₃ content of less than 0.01 g. per liter. In the presence of alkali, the SO₃ concentration of the saturated lime-gypsum solution may be several grams per liter, depending upon the alkali content. However, the effect of alkali on a solution saturated with respect to lime and sulfoaluminate, after the gypsum has become depleted, is minor. For alkali contents normally encountered in portland cement the SO₃ content of such a solution will not exceed 0.07 g. per liter. Thus, if the extract obtained from the hardened paste or mortar contains in excess of 1.0 g. of SO₃ per liter, it is evident that the gypsum was not depleted during the period of curing. On the other hand, if the SO₃ concentration is less than 0.07 g. per liter, it is evident that the gypsum was depleted. The following procedure was used:

Five hundred grams of paste or mortar were placed on clean glass plates and cured at 70 F. and 100 per cent relative humidity. At 18 ± ½ hr. from the time the mixing was completed about one-half of the hardened paste or mortar was removed from the glass plate and pulverized in a porcelain mortar until it all passed the No. 20 sieve. A 200-g. portion of the pulverized material was then placed in a 250-ml. beaker and 50 or 100 ml. of distilled water added.[7] The slurry was then thoroughly mixed with a stirring rod and allowed to stand for 10 min. with occasional stirring. The liquid was then filtered off on a Büchner funnel with the aid of suction until sufficient liquid for the analysis had been collected. If the first filtrate was turbid, it was poured into the original filter bed and drawn through the filter a second time, or it was refiltered with a new paper, without the aid of suction.

Twenty milliliters of the clear extract were transferred to a 400-ml. beaker and diluted with distilled water to 250 ml. Five milliliters of concentrated HCl were added, the solution heated to boiling, and 10 ml. of a hot 10 per cent BaCl₂ solution added slowly (drop by drop) from a pipet. The boiling was continued for 10 min.; then, the solution allowed to stand overnight.

After the precipitate was filtered and washed, the paper and contents were placed in a weighed crucible and slowly ignited until the paper was charred and consumed without flaming. After ignition the BaSO₄ was cooled and weighed. A blank determination, following the same procedure and using the same amount of reagent, was used to correct the results obtained in the analysis. The SO₃ contents were calculated to the nearest 0.01 g. per liter of solution. The weight of BaSO₄ corrected for the blank, multiplied by 17.15, gives the weight of SO₃ in grams per liter.

The averages of the values reported by the several laboratories are shown in Table II. Illustrative plots of the data are shown in Fig. 6. Generally speaking, the tests on the mortars made from the different cements seem to show slightly better agreement between laboratories than do the results on the neat cement bars.

Inasmuch as the equilibrium SO₃ concentration of a solution saturated with respect to calcium hydroxide and calcium sulfoaluminate, after the gypsum is depleted, is less than 0.1 g. of SO₃ per liter, a performance test might require that an 18-hr. extract yield more SO₃

[7] One-hundred ml. of water used with neat cement paste. Fifty ml. of water used with mortar.

Cement	Type	For Least Contraction	For Least Expansion	For Greatest Compressive Strength	Greatest Flexural Strength— One Laboratory	Optimum Heat Generation	Average All Criteria	Indicated from Extraction Test
No. 1	Type I, high C_3A high alkalies	3.5	3.0	3.5	3.0	?	3.5	3.0
No. 2	Type I, high C_3A low alkalies	3.0	2.5	2.5	2.0	3.0-2.5	2.5	3.0
No. 3	Type II, high alkalies	3.0	2.5	2.5	2.5	?	2.5	2.5
No. 4	Type II, low alkalies	2.0	2.0	2.0	2.5	?	2.0	2.0
No. 5	Type III, high alkalies	4.5	3.0	4.0	4.5	?	4.0	3.5
No. 6	Type III, low alkalies	3.5	2.0	3.0	3.5	?	3.0	3.5
No. 7	Type IV, low C_3A high alkalies	3.5	3.5	3.0	3.0	?	3.5	3.5
No. 8	Type IV, low C_3A low alkalies	1.5	2.0	2.5	1.5	?	2.0	2.0
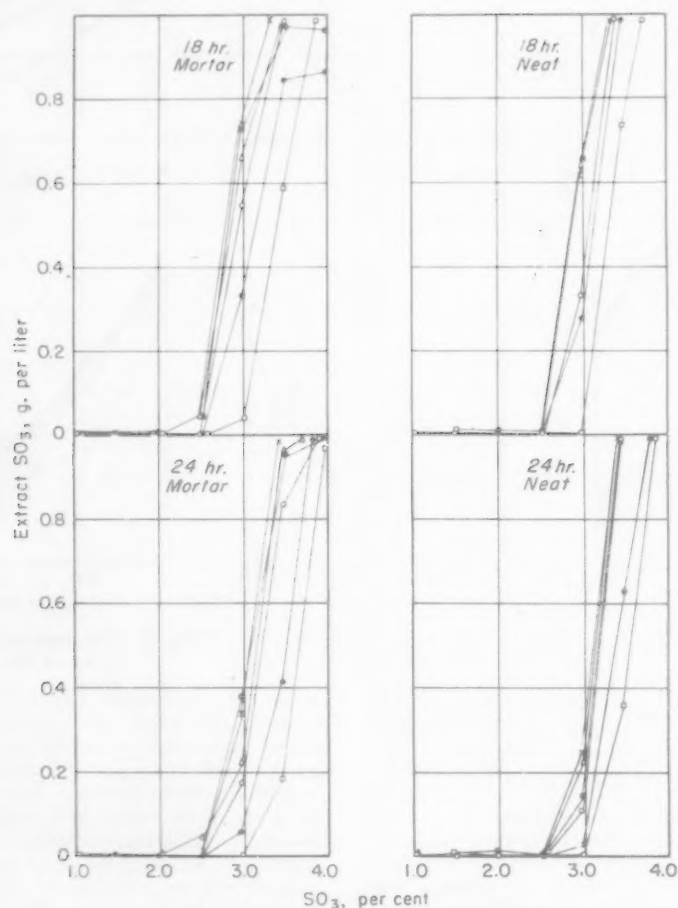
Fig. 6.—Extraction Tests of Cement No. 2.

than 0.1 g. per liter, indicating that the gypsum has not been completely consumed. Similarly, a 24-hr. extract could be required to yield less than 0.1 g. of SO₃ per liter, indicating that excess gypsum is no longer present. However, the results reported by the individual laboratories indicate that the test is not sufficiently reproducible for such narrow limits. More realistic limits could require in excess of 0.2 g. per liter at 18 hr., and less than 0.5 g. per liter at 24 hr. Using these limits would select an optimum, as shown in Table III, which would correspond very closely with the average obtained from the criteria tests.

While the agreement between laboratories was not all that could be desired, it appears that the extraction test is the most promising method of defining optimum gypsum in a specification. Con-

siderable thought has been given to means of reducing variables in the procedure. For example, temperature control of the extraction process has been suggested. However, the work of W. C. Hansen and E. E. Pressler on "Solubility of $Ca(OH)_2$ on $CaSO_4 \cdot 2H_2O$ in Dilute Alkali Solutions,"[8] indicates that temperature is not critical during the extraction. Close control of temperature during the curing period is, of course, important.

Durability Tests.—Three of the participating laboratories conducted durability studies. In the absence of any standard procedure for this work, each cooperating laboratory was requested to study durability in accordance with its usual method. As might be expected, the procedures varied widely, so that it is difficult to compare the results. However, an indication of the effect of gyp-

sum on durability is shown in Fig. 7, which gives the results reported by two of the laboratories. The third laboratory incorporated an air-entraining agent in the mixes, so that the results are not comparable.

One laboratory, No. 3, fabricated durability specimens for each SO₃ content, permitting the curves shown in Fig. 7 to be based on the entire series. Laboratory No. 1 used two SO₃ contents for each cement, one at optimum and the second at the present A.S.T.M. limits. In those cases where the optimum was at the present A.S.T.M. limit, the SO₃ contents were taken at 0.5 per cent above and below such limit. In drawing the curves between the two points (for each cement) reported by laboratory No. 1 consideration was given to the data shown for laboratory No. 3.

The optimum SO₃ content (shown by heavy arrows in Fig. 7), as defined by the criteria tests, is slightly above the SO₃ content for maximum durability. In the presence of air-entraining agents, however, the differences in durability due to varying the SO₃ content appear to be negligible.

In the majority of the results obtained in this study, the cements of high-alkali content showed better durability than did the low-alkali cements of the same type. However, by referring to Table II, it will be noted that the air contents of the mortars, as determined by A.S.T.M. Tentative Method of Test for Air Content of Air-Entraining Portland Cement Mortar (C 185 – 47T)[9] show that the high-alkali cements of types I, II, and IV entrained considerably more air than did the low-alkali cements of these types. Therefore, it seems probable that the relative air contents explain at least a part of the greater durability shown by the high-alkali cements. In any case, the high- and low-alkali cements of each type are not companions differing only in alkali contents.

CONCLUSIONS

The results of this study are summarized in Table III. Giving most weight to the optimum established by

[8] *Industrial and Engineering Chemistry*, Vol. 39, p. 1280 (1947).
[9] 1947 Supplement to Book of A.S.T.M. Standards, Part II, p. 229.

Type I Cements Type II Cements

Low Alkali Optimum High Alkali Optimum Low Alkali Optimum High Alkali Optimum

High Alkali

Low Alkali

High Alkali

Low Alkali

SO₃ Content, per cent

Type III Cements Type IV Cements

Low Alkali Optimum High Alkali Optimum Low Alkali Optimum High Alkali Optimum

Low Alkali

High Alkali

High Alkali

Low Alkali

Cycles of Freezing and Thawing

SO₃ Content, per cent

Curing Conditions and Criteria of Failure

Lab. No. 3 – 28 days Fog
o———o High Alkali Cement – Cycles to 25 per cent Weight Loss
•———• Low Alkali Cement – Cycles to 25 per cent Weight Loss

Lab. No. 3 – 14 days Fog and 250 days Dried
△– – – –△ High Alkali Cement – Cycles to 25 per cent Weight Loss
▲– – – –▲ Low Alkali Cement – Cycles to 25 per cent Weight Loss

Lab. No. 1 – 14 days Fog, 14 Dried and 3 days Water
□——–·□ High Alkali Cement – Cycles to 0.1 per cent Expansion
■——·■ Low Alkali Cement – Cycles to 0.1 per cent Expansion

Fig. 7.—Effect of SO₃ Content of Cement on the Durability of Concretes Made with Various Cements.

compressive strength, expansion, and contraction, an average for all criteria is shown in the next to the last column. This average has been placed at the nearest 0.5 per cent, by inspection only, since it was not considered justifiable to compute an arithmetical average to any decimal figure. The optimum fixed by the extraction test is shown in the last column for comparison. It will be seen that this value agrees with the average of the various criteria for half the cements, and disagrees with the remainder by only 0.5 per cent SO₃.

In every case the high-alkali cement needs more gypsum than the low-alkali cement of the same type, the difference being as much as 1.5 per cent SO₃. Type III cement with a high-alkali content requires the greatest amount of gypsum at the optimum. Next was the type I high-alkali cement, although, surprisingly, type IV high-alkali also seems to require almost as much gypsum to provide the optimum. The data agree with Lerch's conclusion 'that "With cement of low-alkali content, those of high tricalcium aluminate content require larger additions of gypsum than those of low tricalcium aluminate content." Type II and type IV low-alkali cements do not have as high an optimum SO₃ content as type I and type III low-alkali cements.

It is evident from these data that, if advantage is to be taken of the improvement which optimum gypsum will afford, specifications must permit higher SO₃ limits than presently allowed. Although recent changes permit the use of more gypsum with high C₃A cement, these amendments have not been liberal enough to allow the manufacturer full opportunity to use gypsum in the quantity that his particular clinker may require. Increases of 1 to 2 per cent in present limits are indicated. There is ample evidence in these tests that cements of greater volume constancy can be obtained by using an optimum amount of gypsum. Unless the optimum amount is greatly exceeded, there appears to be little danger of excessive expansion which is apparently the only reason for maximum limits on SO₃.

FUTURE WORK OF THE COMMITTEE

The extraction test appears to be the most promising method of defining optimum gypsum in a specification. A modified procedure, in which the paste was continuously agitated at an elevated temperature during the curing period has been tried. Although there was a considerable advantage to the elimination of the grinding step, the results did not show sufficient agreement between laboratories to warrant additional study. The Working Committee is now proposing that the 24-hr. extract test be used to establish the maximum amount of gypsum permitted in portland cement. Further consideration is being given to the 18-hr. extract test for establishing the minimum gypsum content.

Acknowledgment:

Effects of Certain Addition Agents Upon the Frictional and Wear Characteristics of Powder Metallurgy Bronzes

By J. H. Dedrick[1]

Within the past fifteen years powder metallurgy friction materials have attained a prominent position in the heavy-duty brake and clutch fields. These materials generally are run against opposing surfaces of steel or cast iron and though the value of the coefficient of friction depends upon the operating conditions, values in the range 0.25 to 0.50 are acceptable. Wear rates as high as 0.010 in. per hr. as determined by specific tests are considered satisfactory.

The powder metallurgy materials used today are essentially alpha bronzes to which other components are added in varying proportions to produce desirable friction and wear characteristics. Some of the addition agents are silica, graphite, molybdenum sulfide, and lead. A typical parts-by-weight composition used today is as follows: 93 copper, 7 tin, 9.8 graphite, 10.6 lead, and 6.1 silica. The specific function of each of the addition agents has seldom been ascertained. As a result, the lack of information on the influence of such additions, in regard to coefficient of friction and wear rates, seriously handicaps design and applications of such products.

These bronze-base powder metallurgy friction materials are produced by (1) mixing the various ingredients in powder form in special devices, (2) pressing the composite mixture to the desired shape and size in hydraulic presses under pressures ranging from 11 to 15 tons per sq. in., and (3) sintering the pressed pieces in electric furnaces in a $CO-CO_2$ atmosphere at a temperature of around 1450 F.

The literature on powder metallurgy bronze friction materials is slight. No quantitative data are available on the specific influence of lead, graphite, silica, or molybdenum sulfide on the coefficient of friction or on the wear characteristics.

Kuzmick,[2] for example, states that lead acts as a lubricant, in the sense that under certain operating conditions the lead melts and forms a film. He also

states that silica is added to increase the coefficient of friction while graphite functions primarily as a friction reducing agent through lubrication.

Batchelor[3] says that graphite is added in varying quantities as a lubricant, and goes on further to say that silica, alundum, and carborundum are "friction augmenting agents."

Similar results of a highly general nature may be found in the patents on the subject. Fisher's patent[4] describes a power metallurgy friction material. A typical composition given in the patent is 77 per cent copper, 8 per cent tin, and 15 per cent lead, to which five parts by weight of graphite and three parts by weight of silica are added. The coefficient of friction range obtainable is said to be 0.25 to 0.50 depending upon variations in composition.

This work represents an analytical study to determine the specific effect of addition agents to a basic powder metallurgy bronze, on the coefficient of friction and wear rates, when running against steel. To allow for an orderly study, primary components made from powders or otherwise have been tested. In addition, binary and ternary combinations have been studied in order to interpret the behavior of the more complex compositions now used industrially.

MATERIALS, EQUIPMENT, AND PROCEDURE

The primary information desired at the outset of this work was the effect of controlled additions of lead, graphite, silica, and molybdenum sulfide on the friction and wear characteristics of a basic powder metallurgy bronze being run against a steel opposing surface. From a friction standpoint this included solid lubricants such as graphite and molybdenum sulfide, a metallic lubricant such as lead, and an alleged friction-producing agent such as silica. As the work progressed it was deemed advisable to test certain primary components made from powders or otherwise, along with certain binary combinations, in order to make for a more orderly story.

In order to compare the effects of the third component added to the powder

metallurgy copper-tin base materials on an equal volume basis, it was necessary to add the third component in amounts proportional to its density. For example, for graphite and molybdenum sulfide comparisons on an equal volume basis, 2.13 times as much molybdenum sulfide as graphite was added, since the ratio of the densities of molybdenum sulfide to graphite is 2.13. Thus for comparison with a 90 copper - 10 tin - 3 graphite material, a 90 copper - 10 tin - 6.54 molybdenum sulfide composition was used. For all additions to the copper and copper-tin base materials, the minus 325-mesh particle size was used so as to put the surface area factor of the addition agents on an equivalent basis.

A detailed list of the materials studied is given below. The figures given are on a parts-by-weight basis. All powder metallurgy materials were made from elemental powders unless otherwise designated.

1. Elemental Materials (Powder Metallurgy)
   (a) Copper
   (b) Tin (unsintered)
2. Binary Combinations
   (a) Copper-tin (powder metallurgy)
       (1) 90 copper - 3 tin
       (2) 90 copper - 6 tin
       (3) 90 copper - 9 tin
       (4) 90 copper - 15 tin
   (b) Copper-graphite (powder metallurgy)
       (1) 100 copper - 3 graphite
       (2) 100 copper - 6 graphite
3. Ternary Combinations
   (a) Copper-tin-graphite (powder metallurgy)
       (1) 90 copper - 10 tin - 3 graphite
       (2) 90 copper - 10 tin - 6 graphite
       (3) 90 copper - 10 tin - 9 graphite
       (4) 90 copper - 10 tin - 15 graphite
   (b) Copper-tin-molybdenum sulfide (powder metallurgy)
       (1) 90 copper - 10 tin - 6.54 molybdenum sulfide
       (2) 90 copper - 10 tin - 13.08 molybdenum sulfide
       (3) 90 copper - 10 tin - 19.62 molybdenum sulfide
       (4) 90 copper - 10 tin - 32.7 molybdenum sulfide
   (c) Copper-tin-lead (powder metallurgy)
       (1) 90 copper - 10 tin - 15 lead
       (2) 90 copper - 10 tin - 30 lead

(3) 90 copper - 10 tin - 45 lead
(d) Copper-tin-silica (powder metallurgy)
  (1) 90 copper - 10 tin - 3 silica
  (2) 90 copper - 10 tin - 6 silica
  (3) 90 copper - 10 tin - 9 silica
  (4) 90 copper - 10 tin - 15 silica
4. Composite Powder Metallurgy Friction Materials
  (Copper and tin plus two or more addition agents; materials used commercially)
  (a) 91 copper - 9 tin - 16.9 lead - 11.4 graphite - 2.1 unreduced oxide[4]
  (b) 92 copper - 8 tin - 10.2 lead - 6.2 graphite - 2.1 salicylic acid[5]
  (c) 93 copper - 7 tin - 10.6 lead - 9.8 graphite - 6.1 silica - 11.3 iron oxide[6]
  (d) 91.8 copper - 8.2 tin - 17.8 lead - 11.8 graphite - 7.1 silica[6]

In addition to the foregoing, the following nonpowder metallurgy materials were tested:

1. Cast tin.
2. Wrought and annealed copper.
3. Cast and annealed 89.6 copper - 10.4 tin alloy.
4. Electrode graphite.

The powders were mixed by shaking in an Erlenmeyer flask for approximately ten minutes while pressing of the powders into compacts for the wear and friction tests was done at a pressure of 11 tons per sq. in.

The die used was $1^3/_8$ in. in diameter. The bottom plunger of the die was curved so as to produce a surface on one side of the compact comparable to the curvature of the wear machine and friction machine drums.

Sintering was carried out in a $CO-CO_2$ atmosphere. The compacts were embedded in charcoal in a cast iron container and heated to 1450 F. in an electric furnace. In the case of the bronze-base materials, complete alloying of the copper and tin was attained by this practice as evidenced by X-ray diffraction measurements using a "Norelco" Geiger counter X-ray spectrometer.

The nonpowder metallurgy specimens were machined from stock pieces to the desired shape and size for testing. The Swedish sponge iron and cast tin were not heat treated after machining. The copper and bronze specimens were annealed after machining.

Wear tests were carried out by running the specimens against a 14-in. hardened-steel drum, operating at a constant peripheral speed of 1281 ft. per min. The normal pressure on the specimens was 45 psi. which is within the range of commercially used pressures for automotive and tractor brake end clutch applications. The machine was capable of testing six specimens at a

[4] H. S. Williams, U. S. Patent No. 1,738,163.
[5] S. K. Wellman, U. S. Patent No. 2,239,134.

time, and in all cases, duplicate samples were run for each material.

The drum was equipped for internal water cooling, and the water was allowed to circulate for one hour before a test was started. The surface finish of the drum was kept uniform by polishing with emery paper before each test. This produced a surface roughness, in the direction of travel, of 15 microinches (rms.) as registered by an Abbott "Profilometer." This would be considered to be a commercially "smooth" surface.

Before an actual test was started, the drum and specimen surfaces were cleaned with alcohol and ether. The duration of the test was 4 hr. whenever possible. In some instances this time was shortened due to excessive wear rates. Determination of the wear rates was made by a loss-in-thickness measurement of the specimen.

The friction tests were carried out by running two specimens of the same material at diametrically opposite positions, against a 14-in. hardened-steel drum, internally cooled by water, and operated at a constant speed of 1832 ft. per min. The specimens were held against the drum by adjustable tension springs and tests were carried out on a given set of specimens at 17, 34, and 51 psi. pressure on the specimens, in order to study the effect of pressure on friction. A given material was first tested at the lowest pressure and then at the two higher pressures, after which the pressure was again decreased to the two lower pressures. After first "sealing-in" the specimens, the test was carried out at each pressure for approximately 15 min., and observations made as to the constancy of the friction. Unless otherwise stated, the coefficient of friction (μ) figures given in the section on experimental results indicate that the variation in pressure and time used in these experiments had no appreciable effect on the coefficient. The range of pressures used cover those normally encountered in automotive and industrial applications. For example, city bus clutches operate at 15 to 18 psi., while tractor and certain industrial type clutches may operate as high as 40 psi. In some cases it was not possible to carry out this cycle in full due to extreme vibration or too rapid wearing of the specimens.

Preliminary friction tests were carried out on a commercial friction mix at speeds varying from 500 to 2500 ft. per min. with good reproducibility and absence of "fade" from one speed to another. The final speed adopted was based on the fact that it produced the most satisfactory condition from the standpoint of minimum machine vibration.

The tangential frictional force produced by the specimens was counterbalanced by weights through a lever arm system. This relationship was such that when equilibrium was attained between the opposing forces, the coefficient of friction was given by the following formula:

$$\mu = \frac{F \text{ (tangential force)}}{N \text{ (normal force)}} = \frac{2W}{2} \times \frac{1}{\frac{5}{2}S} = \frac{2W}{5S}$$

where S is the spring tension in pounds and W is the counterbalancing weight in pounds.

The same procedure was employed for cleaning the friction drum surface and the specimen surfaces as was used in the case of the wear tests. The surface roughness of the friction drum was the same as that obtained on the wear drum.

EXPERIMENTAL RESULTS

Elemental Materials:

A tabulation of the coefficients of friction and wear rates for elemental materials is given in Table I.

TABLE I.—COEFFICIENTS OF FRICTION AND WEAR RATES FOR ELEMENTAL MATERIALS.

	Coefficient of Friction, μ	Wear Rate, in. per hr.
Powder metallurgy copper	0.86	4.60
Wrought and annealed copper	0.78	2.67
Powder metallurgy tin (unsintered)	0.31[a] 0.30[a]	0.005
Cast tin	0.19[b]	0.006
Graphite	0.26	0.001

[a] 17 and 34 psi. normal pressure on specimens.
[b] 51 psi. normal pressure on specimen.

The results for copper, whether powder metallurgy or wrought, show coefficients of friction and wear rates which are exceedingly high and far outside the commercially acceptable range.

Of particular interest was the fact that the coefficient of friction and wear rate for the copper powder metallurgy material was somewhat higher than for the wrought and annealed copper, indicating that the frictional and wear properties of a given composition material are related to its physical properties. The sintered copper powder material had a density of 7.20 g. per cu. cm., as compared to 8.90 g. per cu. cm. for the wrought and annealed copper.

Both powder metallurgy tin (unsintered) and cast tin showed similar frictional and wear characteristics; their values were considerably lower than those exhibited by copper as is shown in Table I. The density of the pressed and unsintered tin was the same as the cast tin, namely, 7.30 g. per cu. cm. The coefficient of friction for both types of tin were in the commercially and ac-

ceptable range at the lower pressures. Increasing the pressure from 34.0 to 51 psi. resulted in considerable deformation of the specimens, and a drop in friction to an undesirable value.

Solid graphite gave a coefficient of friction of 0.26 which is on the lower side of the commercially acceptable range, and a satisfactory wear rate of 0.001 in. per hr. Thus for certain applications where not too high a coefficient of friction is necessary, it would appear that graphite might be applicable.

Binary Combinations:

A study was made on binary combinations to observe the effect of tin additions to copper in the powder metallurgy state. This was considered to be important since alpha bronzes form the basis of most powder metallurgy friction materials. Also, a cast and annealed bronze was tested for comparative purposes with the powder metallurgy bronzes. Finally, the effect of adding a solid lubricant such as graphite to copper in the power metallurgy state was evaluated to allow for comparisons with later tests on copper-tin-graphite powder mixtures.

The results of the friction and wear tests on the binary combinations are given in Table II.

TABLE II.—COEFFICIENTS OF FRICTION AND WEAR RATES FOR BINARY COMBINATIONS.

	Coefficient of Friction, μ	Wear Rate, in. per hr.
Copper-tin, powder metallurgy:		
90 copper - 3 tin......	0.91	1.19
90 copper - 6 tin......	0.79	0.77
90 copper - 9 tin......	0.67	0.68
90 copper - 15 tin.....	0.57	0.14
Copper-tin, cast and annealed:		
89.6 copper - 10.4 tin..	0.52	0.14
Copper-graphite, powder metallurgy:		
100 copper - 3 graphite	0.56	2.24
100 copper - 6 graphite	0.48	1.38
100 copper - 9 graphite	0.39	1.16
100 copper - 15 graphite...............	0.30	0.28

The addition of tin to copper (powder metallurgy) showed that a decreasing wear rate and a decreasing coefficient of friction may be obtained with increasing tin content. However, in no case within the composition range tested was the wear rate acceptable from a commercial standpoint.

Of interest was the fact that the 89.6 copper - 10.4 tin cast and annealed bronze showed a considerably lower wear rate and coefficient of friction than comparable material (90 copper - 9 tin) made by powder metallurgy methods. This again indicates that internal structure and physical properties have an effect upon the frictional and wear characteristics of a given composition material.

Additions of graphite to copper (powder metallurgy) resulted in decreasing wear rates and coefficients of friction with increasing graphite content. Acceptable friction values were obtained, but the wear rate was much too high in all cases for commercial applications.

Ternary Combinations:

To ascertain the specific effect of sulfide, silica, graphite, and lead upon the frictional and wear characteristics of a powder metallurgy bronze, tests were made on a 90 copper - 10 tin bronze containing each of the above addition agents singly in varying amounts. The results are shown in Table III.

TABLE III.—COEFFICIENTS OF FRICTION AND WEAR RATES FOR TERNARY COMBINATIONS.

	Coefficient of Friction, μ	Wear Rate, in. per hr.
Copper-tin-graphite:		
90 copper - 10 tin - 3 graphite...	0.47	0.265
90 copper - 10 tin - 6 graphite...	0.45	0.237
90 copper - 10 tin - 9 graphite...	0.34	0.002
90 copper - 10 tin - 15 graphite..	0.29	0.002
Copper-tin-molybdenum sulfide:		
90 copper - 10 tin - 6.54 molybdenum sulfide....	0.34	0.037
90 copper - 10 tin - 13.08 molybdenum sulfide....	0.30	0.012
90 copper - 10 tin - 19.62 molybdenum sulfide....	0.26	0.015
90 copper - 10 tin - 32.7 molybdenum sulfide....	0.24	0.010
Copper-tin-lead:		
90 copper - 10 tin - 15 lead......	0.43[a] 0.41[b]	0.123
90 copper - 10 tin - 30 lead......	0.41[a] 0.35[b]	0.059
90 copper - 10 tin - 45 lead......	0.39[a] 0.34[b]	0.058
Copper-tin-silica:		
90 copper - 10 tin - 3 silica......	0.58	0.087
90 copper - 10 tin - 6 silica......	0.40	0.006
90 copper - 10 tin - 9 silica......	0.42	0.002
90 copper - 10 tin - 15 silica.....	0.55	0.005

[a] 17 and 34 psi. normal pressure on specimen.
[b] 51 psi. normal pressure on specimens.

Addition of graphite to a 90 copper - 10 tin powder metallurgy bronze resulted in a decreasing coefficient of friction and wear rate with increase of graphite. When more than six parts of graphite were added, an acceptable wear rate and coefficient of friction was obtained, although the coefficient of friction was rather low. The results obtained are quite different than those obtained on the copper-graphite mixtures were exceptionally high wear rates were obtained in all cases. Thus graphite seems to be much more effective when incorporated in a bronze matrix rather than in a copper matrix. The data in Table III indicate a critical graphite content between six and nine parts by weight to produce acceptable wear and friction characteristics.

The addition of molybdenum sulfide to a 90 copper - 10 tin powder metallurgy bronze also resulted in a decreasing coefficient of friction and wear rate with increasing molybdenum sulfide content. Molybdenum sulfide has a crystalline structure similar to graphite; that is, it has a hexagonal basal cleavage plane and thus would be expected to show lubricating characteristics. In small amounts, molybdenum sulfide appears to be more efficient than the equivalent amounts of graphite in reducing wear and friction. This is shown in Table III where the 90 copper - 10 tin - 3 graphite material may be compared with the 90 copper - 10 tin - 6.54 molybdenum sulfide material. In larger amounts, molybdenum sulfide is more effective in reducing friction but less effective in reducing wear, as compared on an equivalent volume basis with graphite.

The results obtained indicate that bronzes containing more than 13 parts by weight of molybdenum sulfide would have commercial applications, assuming the low coefficient of friction values could be tolerated.

Lead, a metallic lubricant, does not seem to be as effective as graphite or molybdenum sulfide in reducing the wear rate and coefficient of friction of a basic bronze, as is shown in Table III. Addition of 15 parts of lead or more by weight to 90 copper - 10 tin powder metallurgy bronze resulted in an acceptable coefficient of friction, but the wear rates were altogether too high. Higher additions of lead than those shown in Table III could not be incorporated in the bronze due to excessive "sweat out" during sintering. The data do show that at high normal loads the lead-containing bronzes show a lower coefficient of friction, as compared to the coefficient of friction obtained at low normal loads. This is attributed to greater plastic flow of the lead under the heavier normal load, resulting in better film formation on the part of the lead.

Silica, an abrasive, nonfilm-forming agent produced interesting results with respect to friction and wear, when added to a basic bronze. The data in Table III show a decreasing coefficient of friction with increasing amounts of silica up to a certain silica content, and then an increasing coefficient of friction with further silica additions. The wear rate drops progressively with increasing silica content. In all cases the coefficient of friction values are much

higher than for equivalent amounts of lead, graphite, or molybdenum sulfide incorporated in a basic bronze.

Addition of six or more parts by weight of silica produces a satisfactory coefficient of friction and wear rate. However, excessive noise and sparking was encountered during the operation of the silica bronzes which would prohibit their use commercially. Also, some wear occurred on the opposing steel surface.

Composite Materials:

The results on the ternary combinations showed that the maximum obtainable coefficient of friction compatible with a satisfactory wear rate and satisfactory performance (freedom from noise and chatter) was approximately 0.34. This value was obtained for the 90 copper - 10 tin - 9 graphite material. For those conditions of operation where higher coefficients of friction are desirable, it appears necessary to prepare more complex combinations in which silica, graphite, and lead; silica and graphite; or graphite and lead would be incorporated in the basic bronze.

Certain of the commercial powder metallurgy friction materials were tested for friction and wear to directly compare these characteristics with those of the ternary combinations.

Two types of materials were tested—copper-tin-graphite-lead combinations and copper-tin-lead-graphite-silica combinations. The copper-to-tin ratios for these materials were all close to the ratio 90 to 10. The effect of molybdenum sulfide was not studied, but from the results obtained on the ternary copper-tin-molybdenum sulfide materials it would appear worthwhile to make such studies.

The results on the composite commercial materials are listed in Table IV.

It is interesting to note that the material A, consisting of 91 copper, 9 tin, 16.9 lead, 11.4 graphite, and 2.1 unreduced oxides on a parts-by-weight basis showed the exceptionally low coefficient of friction value of 0.24, with an accompanying wear rate of 0.002 in. per hr. The value of the coefficient of friction was approximately equal to that exhibited by pure graphite.

Material B, a 92 copper - 8 tin bronze with 10.2 parts lead, 6.2 parts graphite, and 2.1 salicylic acid on a parts-by-weight basis showed a coefficient of friction of 0.36 and a borderline wear rate of 0.010 in. per hr. Thus by lowering the lead and graphite content, higher friction is obtainable but the wear rate approaches an undesirable value. More desirable wear and friction properties were actually obtained on certain of the ternary copper-tin-graphite combinations.

Some adjustment could probably be made in the copper-tin-lead-graphite composites after rigid experimentation with the relative amounts of lead and graphite required. But from the results obtained on the ternary copper-tin-silica materials it would appear that silica is the most logical material to add if it is desired to increase the coefficient of friction without unduly increasing the wear rate.

The coefficient of friction and wear rate obtained for material D tend to corroborate the reasoning above. This material is comparable to material A except for the addition of 7.1 parts by weight silica. This addition of the silica increased the coefficient of friction from 0.24 to 0.42 and only changed the wear rate from 0.002 to 0.005 in. per hr. The operating performance of the silica bronze was also acceptable from the standpoint of freedom from vibration and noise.

Material C, a copper-tin-graphite-lead-silica composite with some additional oxide present in the form of iron oxide, also showed a high coefficient of friction, 0.48, and an acceptable wear rate of 0.007 in. per hr.

DISCUSSION OF RESULTS

As stated in the introduction, metallic-base friction materials which possess a reproducible coefficient of friction of 0.25 to 0.50 against steels and cast irons and which exhibit low wear ratios are required in practice.

From this standpoint, wrought or sintered copper is not applicable since it exhibits a coefficient of friction and too high a wear rate. Sintered copper shows a higher friction coefficient and wear rate than does the same material in the wrought state, indicating that the physical state of a given material is related to its friction and wear properties. The slightly porous sintered copper might be expected to yield more under a given load than the denser wrought material, thus increasing the area of contact between rubbing surfaces, and consequently the coefficient of friction and wear rate. This hypothesis, however, requires more supporting evidence to be convincing.

Tin, whether in the cast or powder metallurgy state, has been shown to be too plastic for use under operating conditions.

Solid graphite exhibits a coefficient of friction in the lower commercially acceptable range, and a very acceptable wear rate. In certain braking applications where high pressure and prolonged braking are met, asbestos and bronze base friction materials tend to glaze or craze, and it seems that for such applications graphite offers possibilities. This deserves further investigation.

Alpha bronzes are somewhat better than copper from a frictional standpoint but their wear rates are all too high. Additions of tin which produce an alloy beyond the alpha range would permit a lowering of the wear rate, but such applications do not appear to be justified due to the high cost of tin.

Addition of graphite to copper decreases the wear rate and the coefficient of friction. But from all cases studied, the wear rate is still exceedingly high and above the commercial range. It appears that graphite (which produces a wear rate of 0.001 in. per hr. when used alone) is not capable of producing a tenacious surface film when incorporated in copper, since its addition does not cause the expected lower rates to be realized.

Addition of a third component to a basic alpha bronze, in certain cases, however, results in a material exhibiting desirable frictional and wear characteristics.

If more than six parts by weight of −325-mesh graphite or more than three parts by weight of −325-mesh silica are added to 100 parts of a 90 copper - 10 tin bronze, values for coefficients of friction and wear rates are obtained which fall within the commercially acceptable range. This is still true if the amount of graphite or silica added is up to 15 parts by weight.

The interesting effect of silica, in first decreasing the coefficient of friction of a bronze and then increasing it upon further silica addition, might be explained as follows: Small silica additions decrease the area of metallic contact between the opposing surfaces, thus reducing friction. But eventually, as more silica is added, the frictional effect of the silica in ploughing through the steel more than compensates for the reduction in friction due to the decreased

TABLE IV.—COEFFICIENTS OF FRICTION AND WEAR RATES OF COMPOSITE COMMERCIAL MATERIALS.

	Copper	Tin	Lead	Graphite	Silica	Remainder	Coefficient of Friction, μ	Wear Rate, in. per hr.
Material A⁶....	91	9	16.9	11.4	...	2.1 unreduced oxide	0.24	0.002
Material B⁵....	92	8	10.2	6.2	...	2.1 salicylic acid	0.36	0.010
Material C⁸....	93	7	10.6	9.8	6.1	11.3 iron oxide	0.48	0.007
Material D⁸....	91.8	8.2	17.8	11.8	7.1	....	0.42	0.005

contact area, the net effect being an increase in the coefficient of friction.

The low wear rate for the silica-containing bronzes, with accompanying high friction coefficients, may be explained by the hypothesis that some wear was taking place on the opposing steel surface, and also that the silica picked up metal adhering to the steel surface and redeposited it on the friction specimen surface.

The silica-containing bronzes which produce acceptable wear rates have a coefficient-of-friction range from 0.40 to 0.55 which is desirable but one serious objection to their use is that the frictional operation is exceedingly noisy. Also, the graphite additions which insure acceptable wear rates limit the coefficient of friction to approximately 0.34.

Molybdenum sulfide additions are also effective in reducing the coefficient of friction and wear rate of a basic bronze wherein these properties are both above the acceptable range. In small amounts it appears to be more effective than graphite in reducing wear and friction. In larger quantities it is less effective than graphite in reducing wear but more effective in reducing friction. The wear resistance may be attributed to the sulfide's better film-forming properties, yet the greater thermal decomposition of the film as compared to graphite possibly leads to a higher net wear rate. In all cases where molybdenum sulfide was added the wear rate was either too high or borderline, indicating the inadvisability of substituting molybdenum sulfide for graphite.

Lead additions to a basic bronze do not appear to be as effective as graphite or molybdenum sulfide with respect to decreasing friction and wear. The coefficient of friction range obtained for the compositions tried out in this work was between 0.47 and 0.28, but the wear rate was above the acceptable range in all cases.

The results in the ternary combinations indicated that only in the case of the copper-tin-graphite mixtures were satisfactory coefficient of friction and wear rates obtained. The graphite bronzes produced a maximum coefficient of friction of 0.34, however, thus necessitating going to more complex combinations to obtain coefficient of friction in the range 0.34 to 0.50.

The graphite-lead bronze base composite commercial materials studied showed a coefficient of friction range varying from 0.24 to 0.36 depending upon the relative amounts of graphite and lead present and also the total amount of graphite-plus-lead. The coefficient of friction value of 0.24 obtained along with a satisfactory wear rate was lower than that for any of the copper-tin-graphite mixtures tested, and indicates the ability of lead to permit the formation of and anchorage of a continuous graphite film (pure graphite gave a coefficient of friction of 0.25). There appears to be a critical lead-plus-graphite content in order that the foregoing will be true, however, for decreasing the lead-plus-graphite content increased the coefficient of friction to 0.36 and the wear rate to a borderline value of 0.010 in. per hr. Thus a satisfactory high-friction material is not obtainable, in this manner, as a further decrease in lead plus graphite would raise the friction but simultaneously would produce an unsatisfactory wear rate.

Additions of silica in varying amounts to bronze base materials containing graphite plus lead brings coefficients of friction up into the higher acceptable ranges (0.40 to 0.50) with accompanying satisfactory wear rates. Such materials operate satisfactorily from the standpoint of freedom from vibration and noise, in contrast to the bronzes containing silica alone, indicating the necessity of incorporating lubricants. The action of silica on the coefficient of friction and wear rate is what might be expected based upon the results obtained on the ternary copper-tin-silica materials. The reasons for the specific effects of silica in the composite materials may be explained by the same hypothesis as that presented for the copper-tin-silica materials.

CONCLUSIONS

In summation, it has been shown that the coefficient of friction and the wear rate of single metals or metallic phases can be lowered to acceptable amounts through the addition of various other materials. The lowering of the wear rate and coefficient of friction can be attributed to a decrease in the adhesion forces acting between the two metallic friction surfaces, through addition of nonmetallic, nonwelding agents or metallic nonwelding agents. Also, the specific effect of certain commercially used addition agents, in the powder metallurgy friction field, has been ascertained; this should allow a more intelligent approach to their use.

Discussion of Paper on a Simple Field Test for Consistency of Concrete[1]

MR. E. L. HOWARD.[2]—It had been our hope to present in this discussion much data comparing the ball penetration to slump test results. These comparative data have not been gathered because the Kelly Consistency Meter has been so easy to handle and the results so satisfactory that our technicians have stopped using the slump cone.

Two improvements in the concrete inspection using this test have been immediately apparent. First, the speed of the test permits faster adjustment of mixes. Second, the concrete is more uniform because the Kelly Meter is

sensitive to changes in consistency not always detected by the slump test.

The majority of concrete jobs in this area have no inspector testing the material on every pour. Usually the foreman orders concrete at the slump desired by the "eye ball" test. With the Kelly Meter, the servicemen who contact all such jobs can quickly measure the consistency of the concrete at the mixer, in hoppers, in buggies, and in the forms. It has been interesting to note the response of foremen when the ball sinks slowly out of sight in the supposedly 5-in. slump concrete. They have been loud in their praise of the meter and have been quick to use drier concrete.

Since we first began to inspect concrete we have noted the mortar-aggregate separation that occurs from mixer to forms. We have used several methods of test to measure this segregation, but the Kelly ball seems to be the best to find the troublesome spots where the segregation occurs. On a recent job, by making a series of Kelly meter tests, one mixer truck out of several was found to be discharging concrete partly segregated. The trucks dumped into a hopper two at a time. By taking ball penetrations at intervals during the discharging of each truck the faulty one was easily detected. One foreman complained that the delivered concrete was not uniform. Ball penetrations were taken through all handling processes from truck to forms.[3] We found the con-

crete was becoming badly segregated at the last of three collecting hoppers—not at point of delivery as supposed.

Paving job concrete is easily tested for consistency with the Kelly ball. We have tested each batch of concrete mixed as the paver moved along the grade. We have tested the concrete as dumped and at each operation until the finished slab. Inspection is made more effective because of the ease and speed of test.

It is hoped that the ball can be used to control air-entrained concrete on a job large enough to yield conclusive data. To date we have made a few but unrelated tests with air-entrained concrete. These tests do indicate that the ball is sensitive to consistency changes.

We found the ball too heavy for lightweight concrete. We are constructing one that we expect to be workable.

The great advantages in using the Kelly Consistency Meter are the simplicity of operation and accuracy of re-

sults. Because the differences in test results, so common in the operation of the slump cone, are nonexistent in the case of the Kelly ball test, job foremen place more confidence in the inspector. Because consistency measurement can be made with no delay or inconvenience to the job or the contractor's crew, foremen tend to cooperate more with inspection. All in all, better concrete can be and is being made on jobs so controlled.

MESSRS. J. W. KELLY AND NORMAN E. HAAVIK (authors' closure).—It is gratifying that Mr. Howard's widely extended organization has found the use of the ball-penetration test for consistency of concrete to be practical and helpful. Correspondence from a number of other organizations who are trying out the test is also encouraging and so far has revealed no "kinks" to be ironed out. All interested persons are invited to correspond with the authors.

Mr. Howard has consistently found a secondary but important effect of the new test—in many cases the mixes in use have been found to be wetter than necessary, and a stiffer consistency has been agreed upon. It is well known that on many jobs the slump is seldom if ever actually measured, but is judged "by eye." This practice has grown up over the years because of the inconvenience of the slump test; perhaps a contributing factor is the common knowledge that the indicated slump can be made to vary over a rather wide range by differences in condition of the apparatus and in manipulation. When the slump test is made "according to Hoyle," the indications of the ball-penetration test are usually borne out.

Further studies are in progress of possible adaptations of the static ball-penetration test to lightweight concrete, and to a dynamic form of the test which would have sufficient precision for laboratory purposes.

Statistical Properties of Fatigue Data on 24S-T Aluminum Alloy

By A. K. Head[1]

SYNOPSIS

The results of rotating cantilever fatigue tests of 185 specimens of 24S-T aluminum alloy with seven different surface finishes were used to test four hypotheses on the constancy of scatter at different applied stresses. These hypotheses are:

1. That the scatter in values of log N at any particular stress is the same at all stresses for each surface finish.
2. That the scatter in values of applied stress at any particular life N is the same at all N for each surface finish.
3. That the scatter in a direction normal to the S-log N curve is the same at all points on the curve for each surface finish.
4. That the scatter in values of applied stress at any particular life N is the same fraction of the applied stress at all N for each surface finish.

It is shown that none of the four hypotheses given above adequately fits the data. The distribution of values of log N is shown to be approximately normal.

$\mathbf{A}$LTHOUGH it is well known that the fatigue life of a specimen is not absolutely determined by the applied stress but contains an element of statistical variation, not all fatigue experiments are designed to measure this scatter. For many reasons this scatter is important, for example, in comparing the effect of surface finish on fatigue properties, it should be decided statistically if observed differences are real or just accidental because of the inherent

scatter. In the usual experiment no more than two or three specimens are run at each stress. After these data have been plotted an S-N curve is then drawn according to the personal judgment of the experimenter. As has been pointed out by W. Weibull,[2] it may be more useful to test a number of specimens at each stress. In general, for a given total number of specimens, there will be an optimum distribution of specimens among the various stress levels to give the maximum of information on the desired property of the material. For the determination of the endurance limit one such designed experimental procedure has been given by Ransom and Mehl.[3]

Peterson has suggested[4] a procedure for analyzing conventional fatigue data in which only one specimen is run at each stress. In his method he uses a plausible assumption the validity of which will be examined below.
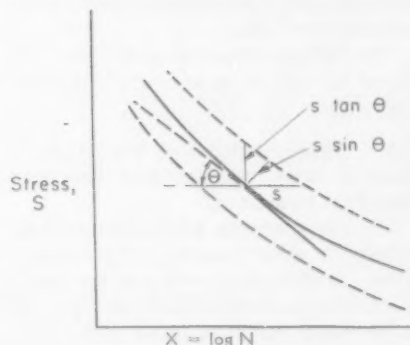
Fig. 1.—S log N Curves.

Systematic fatigue data in sufficient quantity for statistical study are scarce. However, in the course of an investigation of the effect of different methods of preparing fatigue specimens of 24S-T aluminum alloy, extensive data were collected, some aspects of which are presented below.

[2] W. Weibull, "A Statistical Representation of Fatigue Failures in Solids," Transactions, Roy. Inst. Technology, Sweden, No. 27, (1949).
[3] J. T. Ransom and R. F. Mehl, "The Statistical Nature of the Endurance Limit," Journal, Metals Inst., Vol. 1, No. 6, June, 1949, p. 364.
[4] R. E. Peterson, "Approximate Statistical Method for Fatigue Data," ASTM BULLETIN, No. 156, January, 1949, p. 50 (TP 12).

TABLE I.—VALUES FOR x̄ AND s USING VARIOUS FINISHES.

Stress, psi.	400 Grit Longitudinal Polish	Diamond Turned	46 Grit Silicon Carbide	60 Grit Silicon Carbide	60 Grit Aluminum Oxide	120 Grit Cotton Bonded	220 Grit Longitudinal Polish	
46 000	....	....	4	5	5	5	5	n
	....	....	4.851	4.751	4.769	4.743	4.845	$\bar{x}$
	....	....	0.06641	0.02457	0.09811	0.09927	0.1122	s
41 000	6	6	5	5	5	4	4	n
	4.999	4.953	5.125	5.132	5.119	+5.268	5.179	$\bar{x}$
	0.2361	0.2582	0.03442	0.05530	0.06441	0.1598	0.03671	s
36 000	5	5	9	5	7	5	5	n
	5.595	5.495	5.410	5.480	5.411	5.627	5.581	$\bar{x}$
	0.1881	0.1273	0.07837	0.04298	0.06067	0.1408	0.1040	s
31 000	5	4	6	4	5	7	6	n
	6.492	6.279	5.751	5.875	5.776	6.216	6.249	$\bar{x}$
	0.1527	0.2354	0.08782	0.03489	0.09697	0.2238	0.3100	s
27 000	6	....	....	....	....	....	....	n
	7.285	....	....	....	....	....	....	$\bar{x}$
	0.2636	....	....	....	....	....	....	s
26 000	5	5	6	4	5	6	5	n
	7.442	7.280	6.035	6.778	6.275	7.033	7.501	$\bar{x}$
	0.2368	0.1060	0.1698	0.1762	0.2821	0.2033	0.2783	s
24 000	....	....	....	3	....	....	....	n
	....	....	....	7.245	....	....	....	$\bar{x}$
	....	....	....	0.7897	....	....	....	s
21 000	....	....	3	....	....	....	....	n
	....	....	7.369	....	....	....	....	$\bar{x}$
	....	....	0.09772	....	....	....	....	s

EXPERIMENTAL DATA

The data used are taken from an investigation by J. Y. Mann[5] into the effect of surface finish on the fatigue properties of 24S-T aluminum alloy. From the 342 specimens tested a set of 185 were selected which were homogeneous in the following properties:

1. All were tested in rotating cantilever bending at a speed of 12,000 rpm.

2. The hardness of each specimen was measured and lay in the range 125 to 137 Brinell, the mean hardness of each group being in the range 128 to 132.

The number of specimens tested in a group at each stress and surface finish is indicated in Table I.

The 157 results not included were rejected for one or more of the following reasons:

1. Those coming from one batch of material with much lower hardness after heat treatment.

2. Any specimen which fractured at some point other than that of maximum stress. Although the stress at the point of fracture can be calculated, this gives a single result for that stress and could not be used in the analysis.

3. At lower stresses, a test was often discontinued after a large number of cycles without fracture occurred. As the known data for such a group were only a lower limit of its true properties the whole group was rejected.

Data were not rejected for any other reason.

Further details of the experimental procedure and results are given in J. Y. Mann's report.[5]

[5] J. Y. Mann, "The Effect of Surface Finish on the Fatigue Resistance of 24S-T Aluminium Alloy," *Report SM 147*, Aeronautical Research Laboratories (1950).

ANALYSIS

As it is usual to use a semilog plot for fatigue results, the stress S was taken as the independent variable and $x = \log_{10} N$ (where N is the number of cycles to fracture) as the statistically dependent variable.

For each group the values of x, the mean

$$\bar{x} = \frac{\Sigma x}{n}$$

and the estimated standard deviation

$$s = \sqrt{\frac{\Sigma(x - \bar{x})^2}{n - 1}}$$

(where n is the number of specimens in the group) were calculated. The values of $\bar{x}$ and s for each of the groups are set out in Table I.

VARIATION OF SCATTER

The data were used to test four simple hypotheses as to how the scatter (as measured by the standard deviation) varies from point to point along the S-log N curve.

These are:

H1. That for any particular finish the standard deviation s, of x at constant S, is the same for all S. Speaking graphically, this implies that the width of the scatter band in the x direction is the same at all values of S. A statistical test has been given by Bartlett[6,7]

[6] M. S. Bartlett, "Properties of Sufficiency and Statistical Tests," *Proceedings*, Roy. Soc., Series A, Vol. 160, p. 268 (1937).

[7] M. G. Kendall, "The Advanced Theory of Statistics," Vol. II, C. Griffen and Co., London, p. 299 (1946).
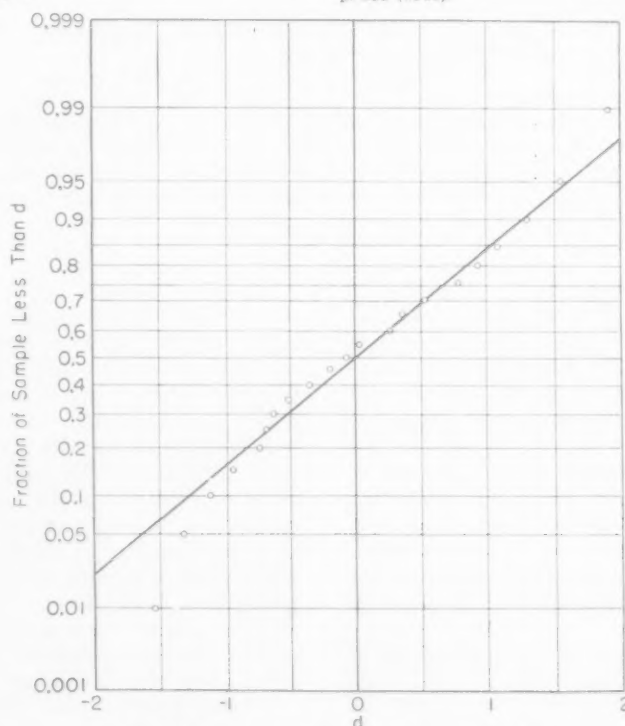
Fig. 2.—Cumulative Frequency Distribution.

TABLE II.—VALUES OF χ^2 FOR THE FOUR HYPOTHESES.

Surface Finish	ν	χ^2			
		H1	H2	H3	H4
400 grit	4	1.36	6.16	3.32	2.30
Diamond turned	3	4.04	11.28*	8.04	7.43
46 silicon carbide	5	9.84	8.37	10.08	11.36*
60 silicon carbide	5	51.01**	9.16	17.94**	16.92**
60 aluminum oxide	4	15.11**	5.54	10.13*	10.69*
120 grit	4	3.00	1.78	1.75	2.23
220 grit	4	13.68**	7.30	8.71	8.43
Total	29	98.04**	49.59**	59.97**	59.36**

for testing the homogeneity of such a set of variances (variance being the square of the standard deviation). The results of Bartlett's test as applied to each of the finishes is given in Table II.

The values of χ^2 given there are a measure of the goodness of fit of the data to the hypotheses, large values of χ^2 indicating poor fit. A single asterisk (*) indicates that if H1 is true this value of χ^2 or larger has a probability of occurrence of less than 1 in 20; a double asterisk (**) indicates a probability of less than 1 in 100. The number of degrees of freedom ν appropriate to each value of χ^2 is one less than the number of groups in the particular finish.

An over-all test of the hypothesis is had on adding the values of χ^2 for all finishes, the total likewise being a value of χ^2 with degrees of freedom equal to the sum of those of the separate finishes. As indicated, this total is extremely improbable and the conclusion is that H1 is extremely unlikely to be true in regards to 24S-T with these finishes.

H2. That the width of the scatter band in the S direction is the same at all values of x. To test this accurately it would be necessary to have data on the scatter in S of specimens which failed after exactly the same number of cycles. Although this is impossible to do experimentally, a convenient approxima-tion is readily obtainable from the present data.

As indicated in Fig. 1, the standard deviation in the S direction is approximately $s \tan \theta$, where s is the standard deviation used above and $\tan \theta$ the gradient of the S-log N curve. The values of θ were measured from the mean curves drawn by J. Y. Mann[5] and so depend to a certain extent on personal judgment.

The values of $s \tan \theta$ so obtained were tested for homogeneity by Bartlett's method and the results given in Table II.

H3. That the scatter band is of constant width normal to its direction. As indicated in Fig. 1 this implies the homogeneity of the values of $s \sin \theta$ and involves the same approximation as the second hypothesis.

H4. Peterson[4] assumes for purposes of analysis that the scatter in the S direction is the same fraction of the mean stress for all values of life x. This can be tested by applying Bartlett's test to $(s \tan \theta)/S$. The values of χ^2 so obtained are indicated in the final column of Table II.

It will be seen that it is very unlikely that any of these four hypotheses is true for 24S-T with those surface finishes tested. It follows that it is very doubtful if Peterson's method for combining fatigue data from several stress levels can be applied to 24S-T aluminum alloy.

FREQUENCY DISTRIBUTION

Most statistical tests for small samples are only exact for data which is "normally" distributed. To obtain an indication of the distribution of fatigue data the following procedure was adopted.

For each specimen the quantity

$$d = \frac{x - \bar{x}}{s}$$

was calculated where x is the log of the life of the specimen, $\bar{x}$ the mean of the group of specimens of the same finish and run at the same stress, and s the standard deviation of the group. If the data are normally distributed, then d is normally distributed with zero mean and unit standard deviation, and all 185 values of d can be used together to form a frequency distribution.

The resulting cumulative distribution is shown in Fig. 2, together with the theoretical straight line for the corresponding normal distribution. The coefficient of skewness is $g_1 = 0.234$ which is not significant.[8] The coefficient of kurtosis,[9] however, $g_2 = -0.877$ is significant. This can be seen in Fig. 2, as extreme values which do not occur as frequently as would be predicted by a normal variable, this effect being common with physical data.

CONCLUSION

For the fatigue tests of 24S-T it has been found that the scatter of values of log N about their mean is an approximately truncated normal distribution. Four simple hypotheses on the variation of scatter between different stress levels are shown to be improbable.

[8] K. Pearson, Tables for Statisticians and Biometricians, Cambridge University Press, p. 224 (1932).
[9] The symbols g_1 and g_2 are those used by G. W. Snedecor, "Statistical Methods," The Iowa State College Press. p. 176 (1941).

Discussion of Paper on Bond Measurement in Reinforced Concrete[1]

Mr. Duff A. Abrams.[2]—During the past 40 years many attempts were made to improve the pull-out test for bond between concrete and steel; the writer made as many such attempts as anyone. The paper under discussion represents a gross underrating of the potentialities of that "well-known procedure."

It is impossible to evaluate the bond tests of the paper, due to omission of fundamental data on quality of cement,

position of bar in molding, condition of loaded end of concrete block, age and number of tests. The paper did not give complete data on a single specimen; Table II and Fig. 5 purported to do so, but gage No. 4 was broken, hence data are useless for scientific study. Parallel readings were obtained for gages No. 5 and 6, but the values were always averaged; this precluded any independent estimate of the concordance of data. In Table IV column headings for end slip should be transposed.

The writer used a rubber cushion in pull-out tests, largely to minimize shocks from slipping grips. Obviously

the authors misunderstood the function of the cushion, since it was used in the the wrong place in a setup without grips. Placing the cushion against the concrete introduced an extraneous factor not met in practice. Expansion of rubber sets up radial stresses in concrete that reduce bond resistance in the lower rings. This can be avoided by placing a planed metal plate of requisite rigidity in direct contact with the concrete. This makes it necessary to mold specimens on rigid, planed metal plates.

The authors were in error in comparing bond resistance of smooth steel tubing with the requirements of the

[1] J. Trueman Thompson and Alvin C. Loewer, Jr., "A New Technique for Bond Measurement in Reinforced Concrete," ASTM BULLETIN, No. 166, May 1950, p. 69 (TP 131).
[2] Consulting Engineer, New York, N. Y.

American Concrete Institute for commercially rolled bars.

The entire "new technique" was based on the erroneous impression that in order to measure elemental bond a uniform bond stress is required.

The pull-out test will give elemental bond simply by noting the loads required to produce first measurable free-end slip, using concrete cylinders 8 in. in diameter and 1-in. bars embedded 5, 6, 8, 10, 12, 15, 18, 21 in. Averages should be based on 8 or more tests made on different days. Plot a curve and extrapolate for 0 embedment. The tests will give also average bond resistance for other free-end slips. A bond-slip curve, similar to Fig. 9 of paper, can then be plotted for 0 embedment.

The above method is not speculation; the writer made such a series of tests 41 years ago (*Bulletin 71*, University of Illinois Engineering Experiment Station) and demonstrated conclusively that elemental bond resistance can be determined experimentally *without producing a uniform bond stress*.

Even if the enormously difficult and costly "new technique" were rid of all of its admitted defects (and other faults not mentioned by the authors) it could not possibly furnish any information that is not obtainable easily and at small cost by the "time-honored" pull-out test.

Messrs. J. Trueman Thompson and Alvin C. Loewer, Jr. (*authors' closure*).—Mr. Abrams calls attention to what he regards as lack of information concerning the specimens, paucity of data, and weaknesses in experimental methods. The paper was the outgrowth of a dissertation in which the information regarding the specimens was set forth. But in keeping with the objective of the paper, which was primarily to present the method of testing, and with the necessities for limiting the length of the publication in ASTM Bulletin, this information was excluded. The same may be said with regard to the volume of data. As to experimental methods, we believe we followed accepted principles. While we take full responsibility for these methods, we did have the advice of Arthur P. Clark of the National Bureau of Standards, whose notable paper, "Comparative Bond Efficiency of Deformed Concrete Reinforcing Bars," which appeared in the *Journal* of the American Concrete Institute, December, 1946, will be remembered. We used the same testing rig design which he originated for his experiments.

We were aware of the extrapolation of slip-bond stress curves for various lengths of embedment used by Mr. Abrams in his experiments published in *Bulletin 71* of the University of Illinois Experiment Station. But we sought to develop a means of making possible the observation of bond strain from its inception to the breakdown of adherence and beyond, into the slip zone. We do not believe that the phenomenon of adherence by materials will be fully understood until it is possible to measure directly the quantities which will enable one to plot stress-strain diagrams for bond. This is admittedly a difficult task, just as is the experimental development of shear stress-strain diagrams. We hope we have made a contribution, (in the interest of more intelligent design,) if in no other way than by directing attention to the desirability of a better understanding of the behavior of adherence in its initial stages.

The Measurement of 60-Degree Specular Gloss[1]

By H. K. Hammond, III[2] and I. Nimeroff[2]

SYNOPSIS

An A.S.T.M. method of test[3] for the 60-deg. specular gloss of paint finishes is based on research done at the National Bureau of Standards more than ten years ago.[4] Until 1949 the method prescribed the illuminator and receiver apertures to be used but gave no tolerances. In order to determine tolerance requirements and to assist the Bureau in determining the requirements for accurate calibration of gloss standards, the rate of change of gloss reading with change of aperture was investigated throughout the gloss scale. Data obtained show that a pair of standards with widely different distributions of reflected light flux are required to check the adjustment of glossmeters for conformance to A.S.T.M. requirements in the increasingly important medium- to high-gloss range.

The Geometric Variables of Glossmeters

In order to investigate the precision to which the many geometric variables of a glossmeter must be specified, an existing goniophotometer was modified to make a versatile glossmeter (see Fig. 1). This glossmeter has provision for controlling accurately (a) the source and receiver apertures, (b) the angles of illumination and view, and (c) the position of the source image relative to the plane of the receiver aperture.

Control of the source aperture is accomplished by employing a pair of condensing lenses to focus an image of the source on an adjustable metallic gate located in the focal plane of an achromatic collimating lens, the focal length of which is accurately known.

The receiver collimating lens also is an achromatic lens for which the focal length is accurately known. It is sufficiently large and so positioned that there is no interception of rays which should reach the receiver. The receiver is precisely located so that the image of the source is brought to focus at the plane of the receptor field stop (receptor window).

Receiver Aperture:

To determine the magnitude of the effect of variation of the rectangular receptor field stop in terms of angular aperture in the plane in which the specular angle is measured, designated α, and in the plane in the polar direction perpendicular to α, designated β, two series of measurements were made on plaques of different gloss with the source aperture held constant at the value prescribed in the A.S.T.M. method. Depolished plaques were used. The results of these measurements are illustrated graphically in Figs. 2 and 3. It will be noted that the measured values for the tiles are a pronounced function of the receiver aperture. On the other hand the measured values for
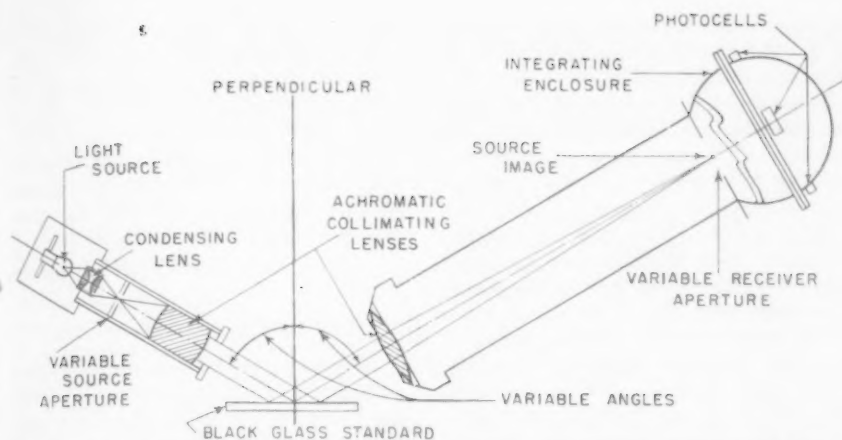
Fig. 1.—The National Bureau of Standards Versatile Glossmeter, Employing Variable Source and Receiver Apertures, Achromatic Collimators, and Variable Angles of Illumination and View.
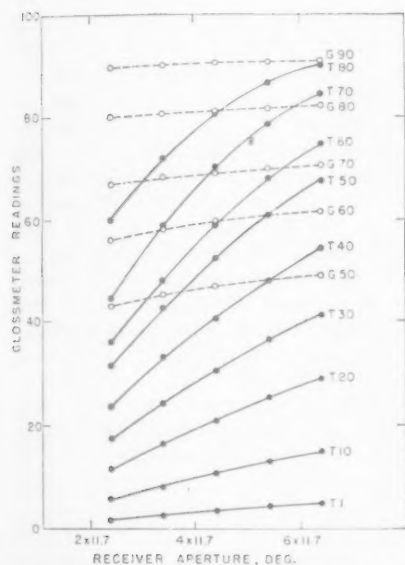
Fig. 2.—Glossmeter Readings Obtained by Varying the Dimension of the Receiver Aperture in the α Plane.

The depolished black glass specimens are designated G followed by the nominal gloss value; the glazed ceramic tile specimens are designated T followed by the nominal gloss value.

the depolished black glass standards remain reasonably constant over the wide range of apertures employed. This is ascribable to the fact that high-gloss tile produces narrow-angle scattering near the specular angle whereas depolished black glass produces wide angle scattering.

Source Aperture:

To determine the effect of source-aperture variation on glossmeter readings, data were obtained on the variation of measured gloss with source aperture with the receiver aperture held constant at the value prescribed in the A.S.T.M. method. The variation of source aperture in the α plane affected the measured gloss of the plaques as shown in Fig. 4. Source-aperture

variation in the plane did not produce as pronounced a difference in measured gloss as did receiver-aperture variation. Variation in the β plane was limited by the size of the source available. Because of the large A.S.T.M. receiver aperture, the source aperture could be increased to only about one half of the size of the receiver aperture. With this limitation the variation of the source aperture in the β plane had a negligible effect.

Position of Source Image:

A properly adjusted glossmeter should form an image of the source centered in the receptor field stop when a flat polished surface is placed in the specimen position. Commercial glossmeters using simple lenses are difficult to ad-
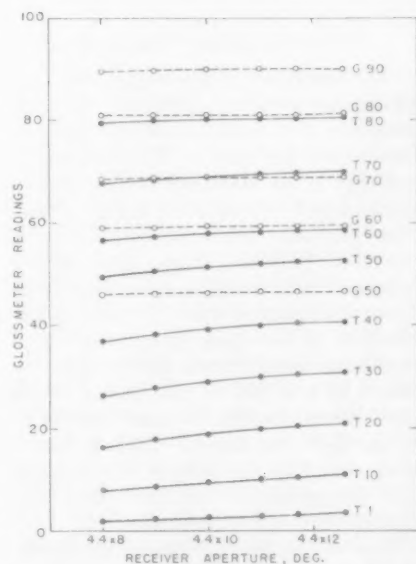
Fig. 3.—Glossmeter Readings Obtained by Varying the Dimension of the Receiver Aperture in the β Plane.

The depolished black glass specimens are designated G followed by the nominal gloss value; the glazed ceramic tile specimens are designated followed by the nominal gloss value.

just because chromatic aberration of the lenses causes light of different wave lengths to focus at different positions. If the red rays are brought to a sharp focus the source image is surrounded by a blue haze. Conversely, if the blue rays are brought to a sharp focus, the image will be surrounded by a red haze. Investigation of the change of measured gloss value with change in the position of the source image shows that the greatest effect is produced on the gloss values of the tile plaques, and that the effect on measured gloss values is not negligible for tile but is practically so for depolished glass. This is explained by the fact that the depolished
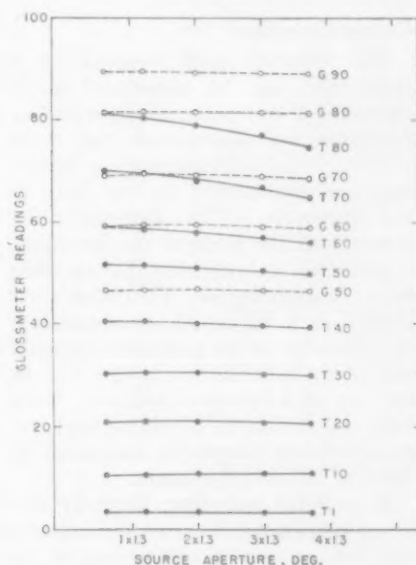
Fig. 4.—Glossmeter Readings Obtained by Varying the Dimension of the Source Aperture in the α Plane.

The depolished black glass specimens are designated G followed by the nominal gloss value; the glazed ceramic tile specimens are designated T followed by the nominal gloss value.

(sand-blasted) glass plaques produce wide angle scattering of some of the light flux while leaving a sharp image of the source, whereas tiles produce narrow angle scattering of nearly all the light flux and thus produce a source image which is not sharp and for which the flux density is varying rapidly across the plane of the receptor field stop. Thus in the latter case changing the focus changes the flux incident on the receiver and consequently the gloss reading. Plaques were measured with the source image focused before and behind the receptor field stop, but no preferred departure of focus from the plane of the receptor field stop was found.

Specular Angle:

Glossmeters are usually adjusted so that the image of the source reflected from a plane reflector, such as that of polished opaque glass, falls on the center

of the receptor field stop. In making this adjustment the angle of incidence may be changed inadvertently so that it becomes 59 or 61 deg. instead of 60 deg. It can be shown from the Fresnel equation[5] that a 1-deg. departure in specular angle at 60 deg. will cause a change of approximately 5 per cent in the specularly reflected flux from a polished surface of refractive index 1.53. However, in the usual method of operation, the instrument is set to read correctly the value of the working standard, then the gloss of the test specimen is read relative to it. If the standard and specimen have nearly the same surface characteristics, no appreciable error will result.

Diffuse Correction:

The reflected light received by a glossmeter may be considered to be composed of two parts: the specular reflection or true gloss contributed by the surface of the specimen and the diffuse reflection contributed by the body of the specimen. The light-scattering property of the body of the specimen, or in a sense its body color, has an effect on the measured gloss. The diffuse component of a high-gloss specimen is a small fraction of the instrument reading and many laboratories ignore it. In the case of a low-gloss specimen, however, this correction should be made especially when comparing specimens of different diffuse reflectances.

A perfectly reflecting, perfectly diffusing specimen reflects no light specularly but causes a "gloss" reading on the instrument because some of the diffusely reflected light reaches the receiver. It has been shown[6] that the fraction, F_r, of the diffusely reflected flux entering the receiver is

$$F_r = \frac{\iota \; \beta \cos \theta}{\pi}$$

where α and β are the aperture angles of the rectangular receiver and θ is the specular angle. For the A.S.T.M. receiver, $\alpha = 4.4$ deg., $\beta = 11.7$ deg. $\theta = 60$ deg., and F_r is computed to be 0.0025. This means that the perfectly reflecting perfectly diffusing specimen having no true gloss, would cause the instrument to read 2.5 if a perfect plane reflector were to read 1000.

Practical surfaces will generally have smaller corrections than 2.5. Except for black surfaces, the correction must

[5] A. Fresnel, "Calcul des Teintes que Polarization Développe dans Lames Cristalisées," *Ann. chim. phys.*, Vol. 17, p. 194–312 (1821).
[6] H. K. Hammond and I. Nimeroff, "Measurement of 60-deg. Specular Gloss," RP 2105, *Journal of Research*, National Bureau Standards, Vol. 44, p. 586, June, 1950.

be made in the low-gloss range in order to grade correctly low-gloss surfaces of different diffuse reflectance. To determine the diffuse correction one must take into account: (a) reflectance of the specimen, (b) departure of practical surfaces from perfect diffusors, and (c) polarization of the source.

With a goniophotometer or a glossmeter in which the illuminator can be rotated about the specimen in the plane of measurement, this correction is most easily evaluated by illuminating the specimen perpendicularly and viewing at an angle of 60 deg. Then the area illuminated is one half of that previously illuminated, but the flux density is twice as great; so the diffuse correction is obtained directly. On the other hand, if 60-deg. illumination and perpendicular viewing are used, the instrument reading will have to be halved to obtain the diffuse correction, because the projected area for perpendicular view is twice that for 60-deg. view.

On the master instrument (Fig. 1) at the Bureau it was found that magnesium oxide, which is considered the practical approach to the perfectly reflecting, if not perfectly diffusing surface, has a diffuse correction of 2.1 This value was obtained numerous times and though not vastly different from 2.5 certainly gives a larger discrepancy than could be accounted for by experimental error. This discrepancy has been accounted for by allowing for the fact that MgO surfaces are not perfectly diffusing and that lamp filaments of the type normally employed cause the incident light to be partially polarized.

GLOSS SCALE AND STANDARDS

Any assembly of apparatus such as a glossmeter requires calibrating and the adoption of a scale on which measurements will be based. The theoretical specular gloss standard is the perfect mirror which is assigned a value of 1000. The practical primary standard is a piece of polished black glass whose specular reflectance is computed from Fresnel's equation when the index of refraction of the glass is known. Such standards usually have gloss values between 90 and 100 on this scale. Black glass is used for this purpose because all of the light flux not specularly reflected is absorbed by the body of the material and the problems associated with diffuse correction are eliminated.

In order to provide means for checking the adjustment of glossmeters and to assist in more accurate measurement

of 60-deg. specular gloss, the Bureau has investigated the suitability of various materials for use as standards and has recently assembled sets of standards for issue. Each set consists of two white Vitrolite glass plaques having nominal gloss values 1 and 90, and eight glazed ceramic plaques having nominal gloss values 10, 20, 30, 40, 50, 60, 70, and 80. The plaques are approximately $4\frac{1}{4}$ in. square.

SUMMARY AND RECOMMENDATIONS

Although photoelectric measurements of specular gloss at a particular angle are convenient for the evaluation of surface appearance, such measurements do not completely evaluate the gloss characteristics, much less the over-all appearance of the surface. In general, the specular gloss of high-gloss surfaces can best be differentiated by observation at small angles from the perpendicular, while matte surfaces can best be differentiated at large angles from the perpendicular. After permissible discrepancies in gloss values have been decided upon the tolerances for the geometric variables for 60-deg. specular gloss can be obtained. On the basis of the data obtained by the authors on the versatile glossmeter and a permissible deviation of ±1 gloss unit for each variable, the recommendations shown in Table I are made.

TABLE I.—RECOMMENDED TOLERANCES FOR PRACTICAL 60-DEG. GLOSSMETERS.

Variable	Recommended Tolerance, deg.
Receiver aperture:	
(1) in plane of measurement	±0.1
(2) perpendicular to plane of measurement	±0.2
Source aperture in plane of measurement	±0.2
Position of source image[a]	(±5 per cent of the focal length of receiver lens)
Specular angle	±0.3 from the specified specular angle

[a] For uncorrected lenses source image should be focused for the circle of least confusion.

The effect of the five variables listed above is cumulative but each is not equally operative over the entire gloss scale. In the high-gloss range where each of the five variables is most effective the instrument reading may be in error by as much as five gloss units if each of the variables happens to contribute errors in the same direction. Such errors may be minimized in practice by employing gloss standards whose light flux distributions are similar to those of the specimens being measured.

Evaluation of Adhesives for Acoustical Tile

By Frank W. Reinhart,[1] Beatrice D. Loos,[1] and N. J. DeLollis[1]

THE properties of commercial adhesives used for bonding acoustical tile to walls and ceilings of rooms were evaluated to obtain data to serve as a basis for the preparation of a specification for these materials. The results of these tests are presented in this paper. A group of 10 adhesives from 8 manufacturers were used in the investigation. The procedures given in Treasury Department Procurement Specification No. 261A[2] were used as a starting basis for the work.

LOADING TESTS

Loading tests were made with a tensile-adhesion type of test specimen at various temperatures and relative humidities for various periods of time.

Procedure:

The tensile-adhesion test specimen was prepared by bonding together two circular unglazed ceramic disks, 4 in. in diameter, with a ⅛-in. thick layer of the adhesive. The adhesive was applied to one disk and then pressed onto the other until the two disks were ⅛-in. apart as indicated by gage blocks. One of the disks had a hook fastened to the center of one side and extending at right angles from the plane of the disk; the load was hung from this hook. The load was also applied in some cases by placing a clamping ring around the disk and suspending the weights from a wire fastened to opposite sides of the clamping ring. The top disk was suspended by placing a clamping ring that had lugs extending in the plane of the disk around the disk. This was then placed on a frame with the lugs supporting the disk. The load was applied immediately by hanging the proper weights from the hook on the bottom disk.

The porous ceramic disks are those used in the Emley Plasticimeter described in A.S.T.M. Specification C 110 – 49.[3] The disks were obtained from the American Instrument Co., Silver Spring, Md.

Various loading tests each consisting

NOTE.—DISCUSSION OF THIS PAPER IS INVITED, either for publication or for the attention of the author. Address all communication to A.S.T.M. Headquarters, 1916 Race St., Philadelphia 3, Pa.
[1] Organic Plastics Section, National Bureau of Standards, Washington 25, D. C.
[2] "Adhesives; Acoustic Material," Treasury Department Procurement Specification No. 261A, March 19, 1940.
[3] Appears in 1949 Book of A.S.T.M. Standards, Part 3, p. 202.

of a cycle of several conditions were used during the course of the project as the work developed. They are as follows:

Load Test	Stress, psi.[a]	Period, hr.	Temperature, ±1 C.	Relative Humidity, per cent
No. 1	0.25	24	21	....
	Nil	24	60	<10
	0.50	24	60	<10
	1.0	24	21	....
	nil	24	21	Immersed in a 0.1 per cent calcium hydroxide solution
	1.0	24	21	....
No. 2	0.25	24	25	50
	0.25	24	60	<10
	0.50	24	60	<10
	0.75	24	60	<10
	1.0	72	60	<10
	nil	24	25	Immersed in a 0.1 per cent calcium hydroxide solution
	1.0	24	60	<10
No. 3	0.25	24	25	50
	0.25	24	60	<10
	0.50	24	60	<10
	1.0	24	60	<10
	nil	72	25	50
	nil	24	25	Immersed in a 0.1 per cent calcium hydroxide solution
	1.0	24	60	<10
No. 4	0.25	24	25	50
	0.25	24	60	<10
	0.50	24	60	<10
	1.0	24	60	<10
	nil	72	25	50
	nil	24	25	Immersed in a 0.1 per cent calcium hydroxide solution
	0.5	24	60	<10

[a] The weight of the disk was negligible when compared to the total weight applied.

When the tests are started at the proper time, the 72-hr. periods come over the weekend. Load test No. 1 is taken from Treasury Department Procurement Specification No. 261A.[2]

One of the problems encountered early in the work was that of cleaning the ceramic disks. This was first attempted by scraping off the excess adhesive, soaking in acetone for at least 24 hr., and drying in an oven at 110 C. for at least 2 hr. This method was not satisfactory; it was observed that resinous material came out of the porous ceramic during the drying period.

The next method used to clean the disks consisted of immersing them in boiling trichloroethylene for 1 hr., rinsing with fresh clean hot trichloroethylene, drying in air for 15 min., and then in an oven at 110 C. for 2 hr. The disks appeared to be clean. However, test results obtained with disks cleaned in this manner were erratic. The porosity was determined by the procedure given in A.S.T.M. Specification D 110 – 49,[3] and it was found that the disks were not clean; they retained only a small fraction of their original porosity.

The cleaning method finally developed consisted of placing the disks in a cold muffle furnace and raising the temperature slowly to 600 C. The heat was then turned off and the furnace allowed to cool to room temperature. Heating or cooling the disks rapidly caused them to crack. The disks were then washed with soap and water, rinsed thoroughly with clean water, and dried at 150 C. This method of cleaning restored their original porosity.

The importance of cleaning the disks properly is shown by the following experiments. Several sets of used disks were cleaned by immersing them in carbon tetrachloride and allowing them to air-dry. Tensile-adhesion test specimens were prepared from these disks with several adhesives and subjected to load test No. 1. All the specimens failed during the cycle in which a stress of 0.50 psi. was applied at 60 C. The disks were then cleaned by heating in the muffle furnace and new specimens prepared with the same adhesives. All the specimens successfully passed load test No. 1.

Attempts were made to use disks of soft pine, which would be used only once and then discarded. It was found, however, that the acoustical adhesives had almost no adhesion to these relatively nonporous wood surfaces.

TABLE I.—TENSILE-ADHESION TESTS WITH ACOUSTICAL ADHESIVES USING LOAD TEST NO. 2.

Test Conditions	Results of Tests[a]									Specimens Passed	Total Specimens Tested
Stress, psi	0.25	0.25	0.50	0.75	1.0	1.0	1.0	nil	1.0		
Period, hr	24	24	24	24	24 (2)	24 (3)	24 (4)	24	24		
Temperature, deg. Cent.	25	60	60	60	60	60	60	25	60		
Relative humidity, per cent	50	<10	<10	<10	<10	<10	<10	Immersed	<10		
N.B.S. Adhesive Identification:											
A					1; 60	2; 0	1; 0		2; 70	1	7
B									2; 50	4	6
C			2; 25						3; 50	4	9
D		3; 20				3; 90			2; 80	none	8
E					1; 60		3; 95			none	7
F					3; 95		2; 75		5; 70	1	8
G		1; 15							3; 20	1	5
H		2; 60	1; 85	1; 60			1; 75			none	5

a The first figure gives the number of specimens failed. The one after the semicolon gives the average per cent failure in adhesion.

TABLE II.—TENSILE-ADHESION TESTS WITH ACOUSTICAL ADHESIVES USING LOAD TEST NO. 3.

Test Conditions	Results of Tests[a]							Specimens Passed	Total Specimens Tested
Stress, psi	0.25	0.25	0.50	1.0	nil	nil	1.0		
Period, hr	24	24	24	24	72	24	24		
Temperature, deg. Cent.	25	60	60	60	25	25	60		
Relative humidity, per cent	50	<10	<10	<10	50	immersed	<10		
N.B.S. Adhesive Indentification:									
B							2; 90	3	5
C		1; 0	1; 0	4; 5				none	6
H							5; 15	none	5
I		1; 5	1; 10	1; 5			1; 40	2	6
J			3; 10	1; 5			1; 5	none	5

a The first figure gives the number of specimens failed. The one after the semicolon gives the average per cent failure in adhesion.

TABLE III.—TENSILE-ADHESION TESTS WITH ACOUSTICAL ADHESIVES USING LOAD TEST NO. 4.

Test Conditions	Results of Tests[a]							Specimens Passed	Total Specimens Tested
Stress, psi	0.25	0.25	0.50	1.0	nil	nil	0.5		
Period, hr	24	24	24	24	72	24	24		
Temperature, deg. Cent.	25	60	60	60	25	25	60		
Relative humidity, per cent	50	<10	<10	<10	50	Immersed	<10		
N.B.S. Adhesive Identification:									
B								6	6
C		3; 5	1; 0	1; 2			1; 85	none	6
D							6; 95	none	6
E							4; 85	1	5
F							2; 95	4	6
G		6; 5						none	6
H				1; 10			3; 30	2	6
I		1; 5						5	6
J				4; 5				1	5

a The first figure gives the number of specimens failed. The one after the semicolon gives the average per cent failure in adhesion.

Results of Loading Tests:

The results obtained in load test No. 2 are shown in Table I. Of the eight acoustical adhesives tested, only one (adhesive B) is considered to be satisfactory on the basis that 50 per cent or more of the test specimens pass the test. Four of the nine specimens made with adhesive C passed the test. Some of the nine test specimens were prepared at a later date than the others, when it was apparent that the adhesive had lost some of the solvent. This preliminary aging or drying as long as it is not too extensive appeared to have a beneficial effect on the strength of the adhesive. Some acoustical adhesives do not form bonds with satisfactory strength after excessive aging.

Examination of the data in Table I shows that the 24-hr. period at 60 C. after the immersion in the calcium hydroxide solution is the most severe. A good acoustical tile adhesive must be unaffected by mild alkaline conditions. Approximately 40 per cent of the failures occurred in this period.

One series of tests was made with load test No. 2 with dial gages mounted in such a manner that changes in thickness of the layer of adhesive could be measured. It was found that the layer of adhesive contracted in thickness. The contraction in the 0.125-in. layer of adhesive ranged from 0.016 to 0.036 in. for the various adhesives. The variation observed with different specimens of each adhesive was also very great. About 90 per cent of the contraction occurred during the first two 24-hr. periods at 60 C. Attempts made to correlate the contraction with the volatile content of the adhesive were unsuccessful. The strongest adhesive (adhesive B) had the greatest contraction and the lowest volatile content.

Another series of tests was made with 5 of the adhesives, using load test No. 3. Since most of the contraction occurred during the first 48 hr. at 60 C., it was decided that the period at 0.75 psi. and 60 C., and 48 hr. of the period at 1.0 psi. and 60 C. could be eliminated. Because of the normal 5-day work week, it was found necessary to introduce a storage period of 72 hr. to take care of the week end. The results obtained in load test No. 3 are presented in Table II. The only adhesive (adhesive B) which was satisfactory in load test No. 2 was likewise the only one which was satisfactory in load test No. 3. It was observed again that about 40 per cent of the failures occurred during the 24-hr. period at 60 C. following the period of immersion in calcium hydroxide solution.

It is apparent from both sets of test results that the immersion in 0.1 per cent calcium hydroxide caused severe degradation of the adhesive bond. Load test No. 4 was designed to decrease the severity of the test by reducing the stress applied in the last period. The stress was reduced to bring it closer to those occurring in service; it is still higher than those encountered in service to obtain an accelerating effect. The results obtained with load test No. 4 are presented in Table III. New batches of adhesive were used in these tests, since it was apparent that the original batches had changed in consistency.

The test results presented in Table III show that adhesives B, F, and I gave satisfactory performance in load test

TABLE IV.—CRACKING TESTS ON
ACOUSTICAL ADHESIVES.

N.B.S. Adhesive Identification	Effect of Drying for 6 Days at 60 C.
A	No evidences of cracking
B	No evidences of cracking
C	Very slightly cracked
D	Badly cracked
E	Badly cracked
F	Moderately cracked
G	No evidences of cracking
H	No evidences of cracking
I	No evidences of cracking
J	No evidences of cracking

No. 4. The improved behavior of the specimens made with these adhesives is attributed to the decreased stress applied in the final period of the test cycle. Adhesive A was not tested because a new batch of the material was not available; the old batch had become hard.

Some of the differences in improved behavior of some of the adhesives in load test No. 4 over that observed in load test No. 2 in the test periods prior to immersion may be attributed to two factors. First, fresh adhesive was used which in most cases gives better results. With older samples from opened cans, the results may be poorer, particularly if the material has become sufficiently dry that it will not wet or flow into the porous surfaces. Second, the disks used to make the specimens for load test No. 2 were cleaned with trichloroethylene while those used to make the specimens for load tests Nos. 3 and 4 were cleaned by heating in a muffle furnace. The first method of cleaning is not satisfactory. However, the differences in behavior of the adhesives in the two tests are not consistent enough to indicate that these two factors caused differences in performance.

CRACKING TEST

Procedure:

Treasury Department Procurement Specification No. 261A[a] has a cracking test which consists of the following. A layer of adhesive, $\frac{1}{8}$-in. thick, is spread on a sheet of glass, 4 by 4 in., and placed in an oven at 60 C. for 6 days. To be considered satisfactory, the adhesive at the conclusion of this period should show no signs of cracking or pulling free from the glass, and it should not be hard or brittle.

Results:

The results of the tests are shown in Table IV. There is no reason to expect the results of the cracking test to correlate directly with the load tests. The effects of a brittle adhesive would be more evident in actual service where relative movements of parts of the building would tend to break the bond.

INITIAL CONSISTENCY

The initial consistency is too soft to make penetration measurements on the test specimens used to determine aging characteristics. Penetration measurements were made on some of the adhesives in the cans when the cans were first opened. This test was made in accordance with A.S.T.M. Method D 217 – 48.[4] The included angle on the cone was 30 deg. The results are as follows:

N.B.S. Adhesive Identification	Depth of Penetration, mm.
B	23.3
E	18.9
F	19.9
G	21.5
H	19.1

CHANGE IN CONSISTENCY ON AGING

A penetration test was selected to determine the change in consistency of the adhesive on aging. With this test it is possible to follow the change in consistency during the aging test by measuring periodically the depth of penetration of a cone. The penetrometer used for this work is described in A.S.T.M. Method D 217 – 48.[4] It should be noted that the depth of penetration readings are inversely proportional to the hardness; that is, the lower the reading the greater the hardness.

Procedure:

Four groups of penetration tests were made. In the first two groups, the cone on the penetrometer had an included angle of 30 deg. while in the last two groups the angle was 120 deg. The weight of the 30-deg. cone used was 104 g. The weight of the 120-deg cone used was 54 g. The specimens for the first two groups consisted of $\frac{1}{2}$-in. thick layers of the adhesives in glass Petri dishes. The specimens for the last two groups consisted of $\frac{1}{8}$-in. thick layers of adhesive on soft pine wood.

The tests were made by spreading the adhesive on the supporting medium, placing the specimen in an oven at the temperatures indicated in Tables V and VI for the periods varying from 24 days at the beginning of the tests to 72 days at the end of the long tests, conditioning at 25 C. and 50 per cent relative humidity for 24 hr., measuring the penetration, and then repeating the heating, conditioning, and measuring cycle until the tests were completed. Each depth of penetration measurement reported is the average of six individual measurements, three on each of two duplicate specimens. The penetration

[4] Standard Method of Test for Cone Penetration of Lubricating Grease (D 217-48), 1949 Book of A.S.T.M. Standards, Part 5, p. 816.

TABLE V.—PENETRATION TESTS ON $\frac{1}{2}$-IN. THICK LAYER OF ACOUSTICAL ADHESIVES DRIED AT 60 C.

N.B.S. Adhesive Identification	Depth of Penetration with 30 deg. Cone, mm.		
	7 days	30 days	45 days
A	10.3	4.5	4.5
B	5.6	2.3	1.6
C	10.4	1.5	0.9
D	13.7	6.1	4.4
E	5.8	0.2	0.0
F	7.9	0.5	0.2
G	14.2	5.7	3.7
H	11.0	6.0	6.0

TABLE VI.—PENETRATION TESTS ON $\frac{1}{8}$-IN. THICK LAYER OF ACOUSTICAL ADHESIVES DRIED AT 60 C.

N.B.S. Adhesive Identification	Depth of Penetration with 120 deg. Cone, mm.		
	1 day	5 days	10 days
A	1.45	0.55	0.40
B	1.00	0.45	0.25
C	1.70	0.35	0.00
D	1.40	0.85	0.80
E	1.05	0.35	0.10
F	1.16	0.55	0.20
G	1.95	1.25	0.95
H	1.80	0.95	0.60
I	1.10	0.35	0.22
J	1.62	0.63	0.28

measurements were reproducible to within ±5 per cent except in a very few cases. Depth of penetration measurements were not made initially because the adhesives were too soft; the penetrator went completely through the adhesive to the supporting medium. The results were plotted as graphs of depth of penetration *versus* time.

Results of Consistency on Aging Tests:

The first group of penetration tests were made at 60 C. The test was run until the penetration values became nearly constant with respect to changes in time; this required about 45 days at the elevated temperature. The general trend of the results is shown in Table V and values for selected periods in Fig. 1. Each curve in Fig. 1 is based on 4 points. The curves in all the figures were drawn by visual judgment to fit the data as closely as possible.

Attempts were next made to accelerate the aging test by drying the adhesives at 60 C. in a vacuum. This was unsuccessful because the materials became filled with gas pockets rendering the specimens unsuitable for the penetration tests.

The second group of penetration tests was made at 75 C. to accelerate the aging effects. The results are shown in Fig. 2. Each curve in Fig. 2 is based on 8 points. It is apparent from the curves in Fig. 2 and from visual observations during the drying period of 21 days that the results obtained on drying at 75 C. are not comparable with those obtained at 60 C.

It is evident that some chemical reactions which occur at a low rate at lower
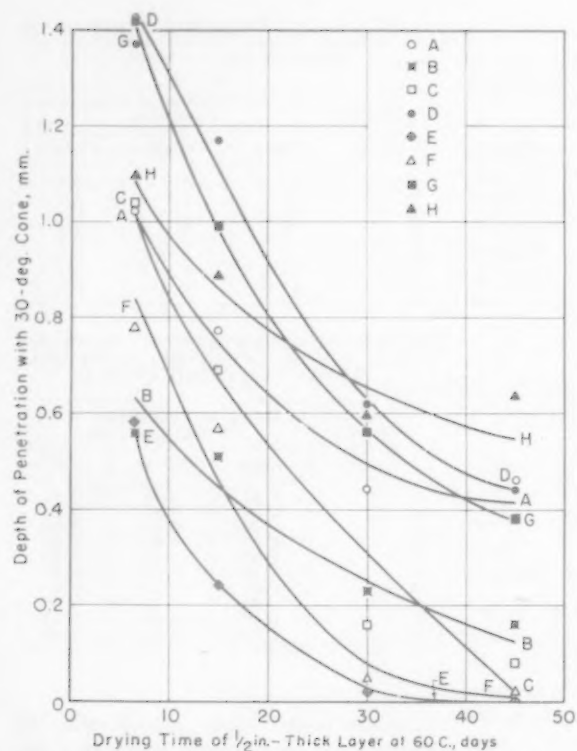
Fig. 1.—Effect of Drying Acoustical Adhesives at 60 C. on Their Consistency.

temperatures are greatly accelerated between 60 and 75 C. It was decided that this temperature, 75 C., was too high to get the desired effects.

The third group of penetration tests were made at 60 C. The thickness of the layer of adhesive on the wood was reduced from ½ in. to ⅛ in., and a 120-deg. cone was used on the penetrometer. These changes were made to reduce the time required to make the test. The time was reduced from 45 to 10 days at the elevated temperature. The

Fig. 3.— Effect of Drying Acoustical Adhesives at 60 C. on Their Consistency.

specimens were aged for 24 hr. at 60 C. then conditioned at 25 C. and 50 per cent relative humidity for 24 hr., and the depth of penetration measured. This cycle was then repeated ten times. The results are presented in Table VI and Fig. 3. Each curve in Figs. 3 and 4 is based on 10 points.

The fourth group of penetration tests was similar to the third group except for the method of measuring the depth of penetration. A question arose concerning the effect of the skin, which forms when the adhesive dries, on the depth of penetration values. To check this effect two sets of specimens were prepared with each of adhesives B, C, and H. Each set of specimens consisted of three individual specimens made by spreading ⅛-, ¼-, and ½-in. thick layers of the adhesives on wood. Two sets of penetration measurements were made. One set was made on the surface in the same manner as all the previous measurements. The other set was made by cutting a triangular in-

Fig. 2.—Effect of Drying Acoustical Adhesives at 75 C. on Their Consistency.
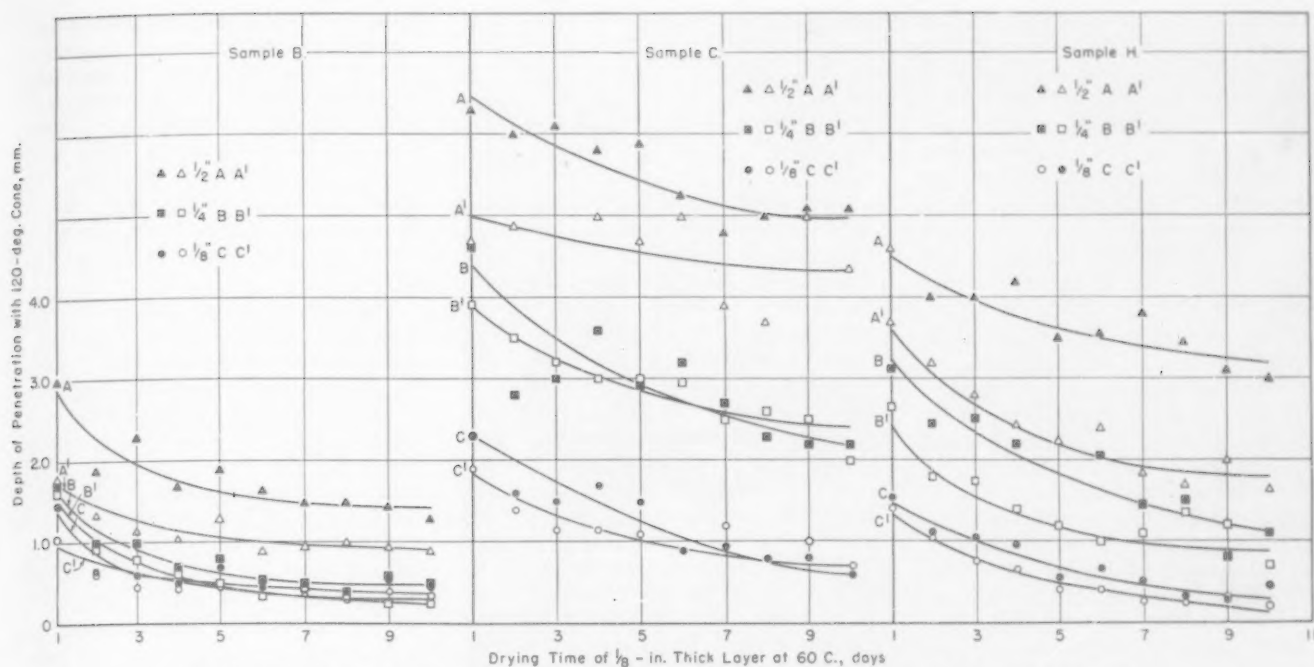
Fig. 4.—Effect of Skin on Depth of Penetration Measurements on Acoustical Adhesive Samples.

cision about 0.3 in. on each side in the skin with a razor blade. The penetration measurement was made approximately in the center of the triangle. The results for samples B, C, and H are shown in Fig. 4. The results show that there is an appreciable difference between these two methods of making the measurements with layers of adhesives $\frac{1}{2}$ in. thick, a slight difference with layers $\frac{1}{4}$ in. thick, and practically no difference with layers $\frac{1}{8}$ in. thick. The difference with $\frac{1}{2}$-in. thick layers of adhesive H is particularly great.

Discussion of Consistency on Aging Tests:

It is difficult to select penetration values suitable for a specification unless they are related to the behavior of the same adhesives in the load tests. It is apparent that the adhesives must be soft enough to be applied readily and still be sufficiently hard to hold the tiles in place initially. On aging, the adhesive must not become so hard that it will crack. A reasonable portion of the flow must be permanent to take care of movements of the building. This latter factor is not evaluated in the load tests

TABLE VII.—VOLATILE CONTENT OF ACOUSTICAL ADHESIVES.

N.B.S. Adhesive Identification	Loss in Weight at 60 C., per cent			
	162 hr.	343 hr.	599 hr.	902 hr.
A.....	6.08	6.53	6.75	6.77
B.....	6.17	6.59	6.75	6.83
C.....	9.48	10.38	10.68	10.71
D.....	6.49	7.01	7.36	7.45
E.....	9.14	9.66	9.87	9.98
F.....	8.86	9.40	9.60	9.66
G.....	7.68	8.38	8.70	8.80
H.....	6.66	7.40	7.76	7.85
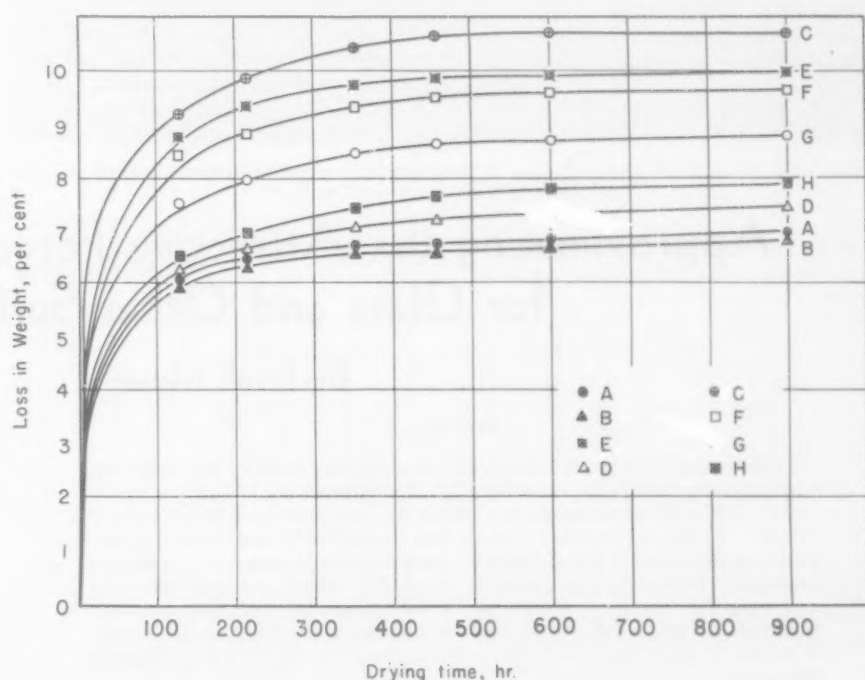
Fig. 5.—Volatile Loss of Acoustical Adhesives, Dried at 60 C.

unless the adhesive hardens, shrinks, and cracks sufficiently to weaken the bond.

VOLATILE CONTENT

The volatile content of the acoustical adhesives was determined by drying a layer less than $\frac{1}{4}$ in. thick in a glass weighing bottle. The stopper was kept on the bottle except when it was in the oven. The materials were dried to constant weight at 60 C. The results are presented in Table VII and Fig. 5.

Each value reported is the average obtained with 3 test specimens.

The volatile content of the acoustical adhesives ranges from about 6.8 to 11 per cent on drying at 60 C. for 902 hr. (about 5.4 weeks). Approximately 90 per cent of this loss in weight occurs during the first week.

SUMMARY DISCUSSION

The satisfactory behavior of an acoustical adhesive in actual service can be judged on the basis of a single

criterion, that is, it must hold the tile to the surface for a long period of time, and over a range of temperatures and relative humidities. The usual period of time suggested by those concerned with the building industry is 20 yrs. It is obviously impossible to make tests for this period of time, and the errors inherent in accelerated service or aging tests make the results of such tests not entirely reliable. These errors are discussed fully in a paper by Reinhart.[5]

The temperatures to which the adhesives are subjected will probably range from 10 to 45 C. and the relative humidities from 10 to 100 per cent. A satisfactory acoustical adhesive must retain sufficient strength at these conditions to hold the tile on the wall. When no changes in composition occur, the strength of the adhesive, like that of all high polymeric organic materials, will decrease as the temperature or relative humidity is increased. This may or may not be true when changes in composition occur as in the evaporation of a solvent from the mixture. The adhesive must remain sufficiently plastic under these conditions and on aging to eliminate failures caused by

[5] F. W. Reinhart, "Degradation of Plastics," S P E News, Vol. 4, No. 7, September, 1948. pp. 3-6, 16, 17.

relative movements of parts of the building.

The acoustical adhesives must also be unaffected by contact with mild alkaline conditions such as may be encountered in some building constructions.

The load tests used in this investigation were made at a temperature higher than those encountered in service to accelerate the aging effects. It appears that a temperature of 60 C. is about as high as can be used. Higher temperatures cause results not normally observed at lower temperatures. In these tests the specimens are immersed in calcium hydroxide solution for one period of the cycle to acclerate the effect of contact with alkaline medium. Load test No. 4 appears to be the most satisfactory of those tested. The periods in this cycle are scheduled so that it will fit the work week of most laboratories, and three out of ten acoustical adhesives passed the test. It should be noted that this test will not evaluate the effects of relative movements of the building.

It would be preferable to select requirement limits for the test on the change of consistency on aging for satisfactory adhesives without reference to any other test method. This could

only be done if sufficient information concerning behavior in service over a period of time was available. Such data are not available. The only method of setting requirement limits at the present time is to judge the results on the basis of the behavior in the load test. In setting the limits, weight must be given to the fact that an adhesive which becomes hard will not be satisfactory in service.

The first group of tests made with ½-in. thick layers of adhesive at 60 C. and a cone of 30 deg. required 45 days for the depth of penetration measurements to become approximately constant. This is too long for a specification test. The tests made at 75 C. did not give satisfactory results. By changing the layer of adhesive to ⅛-in. thick and making the measurements with a 120 deg. cone, the time of the 60 C. test was reduced to 10 days. This latter method appears satisfactory.

The results of the cracking test indicate whether or not an adhesive tends to crack and get brittle when it shrinks and dries.

The volatile contents of the acoustical adhesives are similar. The various manufacturers' products range from about 6.8 to 11 per cent.

Approximating the Attractive Forces of Adhesion for Glass and Other Surfaces

By Frank Moser[1]

SYNOPSIS

Contact angle studies are described for predicting polarity and adhesion. Compatibility of materials is indicated if the materials studied gave approximately the same contact angle in a (otherwise identical) material-air-solvent system. Data are presented to show that compatibility and subsequent adhesion of materials can be secured by using either a heterogeneous adhesive material (with internal compatibility or affinity) which provides both polar and nonpolar functional groups which are compatible with the dissimilar materials, or single-component materials having a polyfunctional molecule with two or more functional groups having selective affinities for dissimilar materials.

MUTUAL attraction is essential between two substances to form an adhesive bond. Whether the adhesive forces are chemical or physical, the basic attractions must be either electrostatic or associated with the orientation-induction effects of molecules and with the forces produced by electronic mo-

tions within the molecule. Such attractions, forces, and effects are reflected at the surface of the material and they influence the surface properties which are important in such phenomena as wetting and adhesion.

To explore further surface attractions, contact angle studies were initiated to determine if any correlations existed among wetting of a surface, orientation of resinous molecules attracted to the surface, and subsequent adhesion between the two components. The presence of unsatisfied forces, or points of

attraction, on the surface of glass and other solids is due to exposed functional groups; that is, the surface arrangement of the molecules in the solid determines the linkage between resinous molecules and the exposed functional groups of the solid surface molecules. The polarity, or surface orientation, may be obtained from contact angle measurements in solid-air-liquid systems using either polar or nonpolar solvents as the calibrating medium.

Experimental:

Wetting of smooth solids and adhesive coated glass surfaces was studied by the captive bubble method (2)[2] which was improved by Wark and Cox (3) and recently modified by Kellogg (4). Figure 1 shows the apparatus constructed for the study. Orientation of adhesive molecules on solid surfaces was approximated by the measured contact angles of clean and coated surfaces in

otherwise identical systems. Figures 2 to 4 show photographic views of the air bubble in contact with the solid.

The angle of contact was obtained by measuring the angle subtended by the tangent at the point of contact of the bubble with the surface. In making the determinations the cross hairs in the eyepiece were laid along the tangent and the angle read directly in degrees.

Compatibility between an adhesive and a surface was assumed if approximately the same angles were obtained for the adhesive coated surface as for the clean surface where the same calibrating medium was used to obtain the contact angles. Polarity measurements were made by immersing the solid in a calibrating liquid of known polarity. Perfect wetting (zero angle of contact) was obtained if the polarity of the solid was identical with that of the liquid, while a finite contact angle established the degree of polarity or nonpolarity.

Viscosity of resinous solutions was estimated with the Brookfield viscometer. Adhesion was tested under tensile loading with a Dillon motorized tester using the tensile specimen and jigs previously described (3). The tensile specimen was a cross-lap type consisting of a bonded area of 1 sq. in. obtained by cementing two pieces of 1 by $1\frac{1}{2}$ by $\frac{1}{2}$-in. glass to each other so that $\frac{1}{4}$-in. free end of glass extends on each of the four sides of the bond in order to hold the specimen in the jig for testing.

Contact angles were determined using glass-air-adhesive solvent systems and adhesive coated glass-air-water systems. Glass is a hydrophilic material giving a zero angle of contact in a glass-air-water system, but a glass surface may be classified also as "organophilic" since wetting occurs when any one of a large number of the common organic solvents is used as the third component.

Wetting:

Surface structure influences attraction between solids and liquid adhesives or thermoplastics. For example, a glass surface exposed to atmospheric conditions consists essentially of exposed hydroxyl groups.[3] Therefore linkage is possible either as an oxygen bond or as a hydrogen bridge. In either case attraction is dependent upon the orientation of the functional group of the resinous molecule. A schematic reaction of the surface molecules of a glass with a dimethyl chlorosilane is illustrated using

[3] A reaction at temperatures lower than 500–600 C. between kaolin and Si—O bonds of the glass surface, or a reaction with an adsorbed water film or with H₂ from the atmosphere might form these OH groups.—Ed.

Fig. 1.—Contact Angle Assembly Showing 20-Power Microscope, Proctractor Glass Cell, Bubble Holder and Sliding Platform.

A to represent the nonreacting portion of the molecule:

Glass surface molecule + Dimethyl chlorosilane molecules → Modified glass surface molecule

The new surface is now very hydrophobic and the contact angle of this "modified" glass surface is approximately 95 deg. in a glass-air-water system indicating that nonpolar ions are present on the outer surface. The change that has occurred in polarity and of wetting at this nonpolar, chlorosilane glass surface seems to explain the fact that most of the resinous materials may be cured as films on it with no apparent adhesion to this surface. All this indicates that both polarity and wetting are important surface properties to consider in obtaining adhesion and, undoubtedly that wetting and subsequent adhesion depend upon the presence of adjacent "compatible" functional groups for joining the two substances.

Another nonpolar solid material, "Teflon," with approximately a 90-deg. contact angle in a Teflon-air-water system exhibits nonwetting properties and there is no apparent adhesion of resinous materials to this material. This further substantiates that surface molecular orientation influences wetting, and, ultimately, adhesion.

Some Results:

Various types of glass were examined in glass-air-water systems; and the contact angles for the phosphate, barium crown, lead glasses, and pure quartz (SiO_2) were less than 20 deg. indicating good wettability. Also, a copper-modified-glass surface, prepared according to a method proposed by Weyl (7) gave a zero angle of contact. Other results (see Table I) show that glass is wetted by aliphatic, aromatic, and chlorinated hydrocarbons, esters, ketones, alcohols, amines, and organic acids. Also, liquid monomers and various plasticizers give

(a) Glass

(b) Buna-S

Buna-N

(d) Natural Rubber

Fig. 2.—Contact Angles of Glass and Rubber Surfaces in Solid–Air–Water Systems.

good wetting as indicated by the contact angles measured on glass surfaces which ranged from 0 to 20 deg.

Table II gives a classification of polarity with respect to contact angles and should be useful in selecting resins for adhesives for various solids. The contact angles for the resinous solids are presented in Table III along with a polarity classification. This classification allows prediction of the compatibility of respective materials for use in adhesive formulations that are compounded for joining glass. As the com-

patibility classification is based on contact angles, the resins listed in Table III that have a similar contact angle, or the same polarity classification letter, should be compatible. Adhesive properties of any two of these resins to a third surface would be predicted by similarity of contact angles (or by having the same letter) obtained for the three components. For example, a plasticized polyvinyl butyral, a Buna N and a Durez phenolic (12987) resin may be used together for joining glass; whereas neither polyethylene nor polytetrafluorethylene shows no adhesion to glass. Other materials with 30 to 40 deg. contact angles give better adhesion to glass if compounded with materials having an angle of contact between 40 and 0 deg. as exhibited by vinyl modified phenolics.

Contact angles of cured rubber stocks are often influenced by small amounts of antioxidants. For example, the addition of small amounts of antioxidant to a natural rubber increased the contact

TABLE II.—POLARITY CLASSIFICATION WITH RESPECT TO THE MEASURED CONTACT ANGLE.

Contact Angle Range	Classification Assigned	Symbol
0 to 20...	Polar	A
21 to 45...	Slightly polar	B
46 to 70...	Moderately nonpolar	C
71 to 100...	Nonpolar	D

TABLE I.—CONTACT ANGLE OF GLASS-AIR-LIQUID SYSTEMS.

Liquid	Contact Angle. deg.
Distilled water................	0
Methyl ethyl ketone.........	0 to 4
Acetone.....................	0
Ethyl alcohol................	0
Ethyl acetate................	10 to 12
Benzaldehyde................	5
Petroleum ether.............	0 to 3
Carbon tetrachloride.........	0
Monochlorobenzene..........	0
Lactic acid..................	10 to 12
Diethylene glycol............	7 to 10
Toluene.....................	0
Xylene......................	0
N-butyl alcohol..............	0
Glycerol.....................	8 to 10
Sulfonamide (MS 80).........	3 to 5
"Rosin amine D".............	0
"Alochlor 1248"..............	14 to 17
Tricresyl phosphate..........	4 to 6
Butyl diglycol carbonate.....	4 to 7
Styrene (monomer)..........	0
Methyl methacrylate (monomer).....................	0
Vinyl acetate (monomer).....	0

(a) Durez 12687 Film

(b) Durez 12897 Film

(c) Chlorinated Rubber Film

(d) Hycar OR 25-1032X1 Film

Fig. 3.—Contact Angles of Adhesive Materials Coated on Glass Surfaces.

(a) Polyblend Film   (b) Durez 12687 and Hycar OR 25-1032X1 Film

(c) Durez 12987 and Chlorinated Rubber Adhesive Film   (d) Durez 12687-Polyblend Film

Fig. 4.—Contact Angles of Adhesive Material and Formulations Coated on Glass Surfaces.

angle of the cured stock approximately 35 deg. A cured Neoprene stock has an angle of 80 to 85 deg., while an uncompound stock (Neoprene G.N.A.) has a 35- to 37-deg. angle in a Neoprene air-water system.

Viscosity Effects:

The solution type of adhesives is not suitable for studying readily the compatibility of an adhesive material with a solid surface since the organic solvent used usually masks the effects of the resin. This is due to the perfect wetting ability of most of the organic solvents.

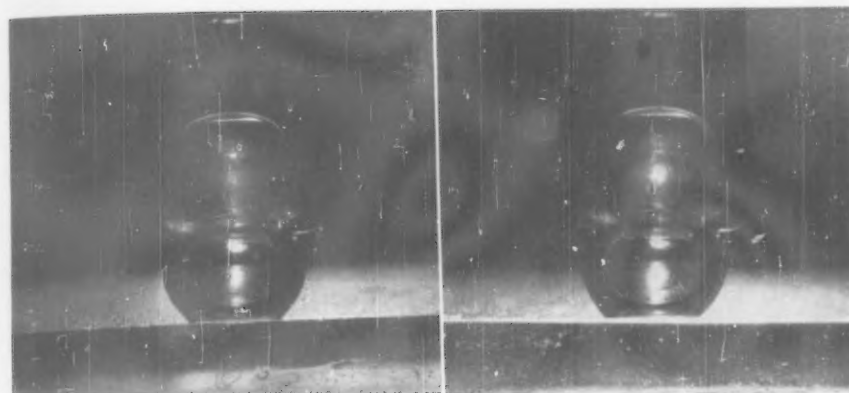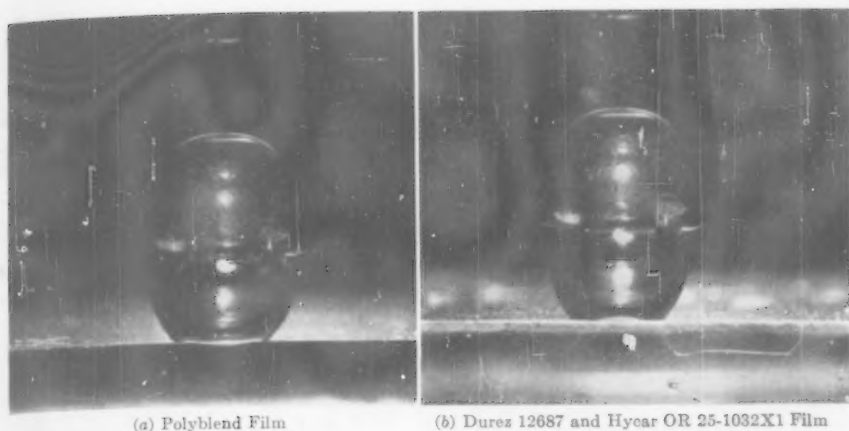trations. Table IV shows the viscosity and contact angles (as an index of wetting) for a polymerized vinyl acetate monomer, a polyvinyl acetate dissolved in a solvent, and tertiary amyl phenol-aldehyde in ethyl alcohol. The results indicated that no appreciable change occurred in wetting with a corresponding viscosity increase. Apparently chemical structure has greater effect on wetting than the viscosity.

Adhesive Formulation According to Contact Angles:

Adhesive formulation was undertaken to explore the postulation that substances having similar contact angles in various solid-air-water systems should be compatible to join materials also having a similar contact angle. If compatible functional groups are not available an adhesive polyfunctional molecule may be prepared having groups compatible with the molecular orientation of each surface of dissimilar materials. In this manner adhesion between two dissimilar materials, through the proper adhesive, is possible.

In Table V the contact angle, bond tensile strength of glass-to-glass assemblies, and water resistance for some ad-

TABLE III.—CONTACT ANGLES AND POLARITY OF SOLID SURFACES IN SOLID-AIR-WATER SYSTEMS.

Solid Surface	Contact Angle. deg.	Classification[a]
Glass...................	0	A
Polymethyl methacrylate............	63	C
Polyvinyl butyral.......	12 to 15	A
Polyvinyl acetate.......	40 to 42	B
Polyvinyl chloride acetate...............	47 to 50	C
Polyethylene...........	90 to 92	D
Polytetrafluoroethylene...	95	D
Polyvinylidene chloride...	35	B
Alkyd styrene..........	33 to 35	B
Cellulose acetate.........	42 to 43	B
Polybutene............	41 to 43	B
Buna-N rubber..........	12 to 15	A
Buna-S rubber..........	62 to 63	D
Butyl rubber...........	40 to 43	B
Neoprene (G.N.A.)......	34 to 36	B
Neoprene rubber (cured stock)............	80 to 82	D
Natural rubber (cured stock)............	30 to 33	B
Pliolite (cyclo-rubber)...	37 to 40	B
Phenyl - aldehyde (Br 9432)............	35 to 38	B
Buna - S (cyclized in H₂SO₄)...........	27 to 30	B
Durez phenolic (12687)...	48 to 50	C
Durez phenolic (12987)...	20 to 21	A

[a] Key: A = polar; B = slightly polar; C = moderately nonpolar; D = nonpolar.

TABLE IV.—EFFECT OF RESIN CONCENTRATION AND VISCOSITY UPON CONTACT ANGLES IN GLASS-LIQUID SYSTEMS.

Resin solution	Concentration, per cent	Viscosity, centipoises	Contact angle, deg.
Tertiary amyl phenol - aldehyde, in ethyl alcohol.......	25	8	0
	50	23	0
	62.5	114	3 to 6
	75	550	3 to 5
Vinyl acetate monomer....	Initial solution	4	0 to 3
	2 h r. after polymerization........	246	0 to 5
	4 h r. after polymerization........	515	0
	8 h r. after polymerization........	1720	0
Polyvinyl acetate, AYAF, in methyl acetate.....	25	214	2 to 4

Therefore, a different approach was used in that viscosity of solutions was varied by polymerization of monomers, such as vinyl acetate, and subsequent measurements were made of viscosity and contact angle at various resulting concen-

TABLE V.—CONTACT ANGLES IN COATED GLASS SURFACE-AIR-WATER SYSTEMS.

Type of Coated Surface	Contact angle, deg.	Tensile strength, psi.	Water resistance
		Adhesion, Glass-to-Glass Surfaces	
Modified phenol	55 to 58	1600	Good
Vinyl phenol...	61 to 64	1250	Good
Modified phenol	55 to 58	1170	Good
Vinyl cellulose..	37 to 39	200	V. poor
Polyvinyl acetate.........	25 to 28	660	V. poor
Acrylate.......	76 to 78	630	V. poor
Vinyl cellulose..	68 to 72	800	Fair
Cellulose-shellac.......	51 to 53	350	Poor
Cellulose-acetobutyrate.....	60 to 63	50	Poor
Neoprene (G.N.A.)....	33 to 35	100	Poor
Neoprene base..	81 to 83	150	Fair
Phenol Buna-N..	47 to 48	1500	Fair
Vinyl butadiene-acrylonitrile..	21 to 23	1200	Poor
Polyvinyl acetate, AYAF..	44 to 46	240	Poor
Polyvinyl chloride-acetate VMCH......	61 to 63	150	V. poor
Polyvinyl butyral (plasticizer)........	15 to 18	1230	Fair
Polyvinyl butyral (no plasticizer)........	33 to 36	600	Fair
Cyclo rubber-copolymer-vinylacetate..	55 to 58	500	Good
Phenyl-aldehyde	72 to 75	[a]	[a]
Carnauba wax..	86 to 88	[a]	[a]
Glyceryl mono-stearate......	88 to 90	[a]	[a]
Methyl chloro-silane........	94 to 96	[a]	[a]

[a] No adhesion.

hesives and pure resins are given. The data appear to show no definite correlations for a number of the commercial adhesives as, usually, the formulations are a mixture of resins or copolymer materials having polyfunctional, oriented

molecules. For example, the vinyl-phenol type listed exhibits good adhesion to glass and metal surfaces with good permanence of bond. In a resin coated surface-air-water system the contact angle of the adhesive coated film was 63 deg. which undoubtedly indicates the resin's good water resistance. However, its bond strength indicates a different orientation of the vinyl-phenol film had occurred when the resin was coated on this surface. The vinyl portion of the adhesive with its lower contact angle, 12 to 15 deg., is attracted to the glass and leaves an exposed phenolic group with a hydrophobic surface. Other adhesives which are composed of bifunctional and trifunctional molecules give a high contact angle and good tensile strength values. Pure materials that gave the higher angle of contact consisted chiefly of nonpolar functional groups having very little attraction to glass. Thus, in using contact angles for predicting adhesive action, the orientation of the adhesive composition must be considered as well as that of the surfaces to be joined.

In formulating an adhesive for a butyl rubber surface to glass, contact angle measurements indicated that butyl rubber is slightly nonpolar, if glass is considered as polar, and has a 42 to 45 deg. angle in a rubber-air-water system in comparison to the 0 deg. angle for the glass-air-water system. Therefore, a bifunctional adhesive will be required which is capable of orientating ts slightly nonpolar end toward the butyl rubber and the polar end to satisfy its affinity for the glass. Such formulation based on these requirements, using Vinylite VMCH and Pliolite S50, was prepared and when the Vinylite-Pliolite film was cured on a glass surface, a 50 to 52 deg. contact angle was obtained in the film-air-water system. This signified that the film surface would be compatible with the rubber. Adhesion tests proved this to be true with adhesion failure occurring from the glass surface. Therefore in coating the glass with a colloidal silicate phenolic type primer, a film surface is produced which has good adhesion to the glass and has an exposed nonpolar surface having a contact angle from 52 to 55 deg. Thus, compatibility is now obtained among the adhesive, butyl rubber, and primed glass surfaces. Subjecting this cured assembly to ten-

sile loading, a bond strength of 300 psi. was obtained with failure in the butyl rubber.

Another illustration of obtaining adhesion for two surfaces is to find a resin or a resinous mixture having the same range of compatibility as the two surfaces. In this case a fully cured Buna-N rubber stock was to be joined to glass or to stainless steel. Studying this combination using contact angle measurements, compatibility seemed assured since in a glass-air-water system perfect wetting (zero angle) was obtained on the glass, a 15 to 18-deg. angle for the Buna-N surface, and a 20 to 21 deg. angle for the Durez phenolic (12987) surface. This bond was evaluated under tensile loading, and an average tensile strength of 690 psi. was obtained with failure in the rubber layer.

The joining of dissimilar materials presents a more difficult problem and requires an adhesive formulation composed either of mixed resinous materials with mutual compatibilities or copolymer molecules with varied functional groups exposed for selective attraction. In either case adhesion is secured by exposed groups of bifunctional heterogeneous molecules that are available for attracting the dissimilar surfaces to the respective portion of the adhesive molecule or mixture of dissimilar aligned molecules through an orientation phenomenon. The above postulation is merely an extension of the De Bruyne hypothesis (1) which contends that polar surfaces attract, while nonpolar surfaces are attracted to nonpolar molecules. If the surface varies in polarity due to different exposed groups, then the resinous combinations or copolymer molecules must be prepared to provide the necessary linkage for attraction of the respective functional groups. A similar postulation was presented by Von Thinius (6) for obtaining adhesion between dissimilar materials, polyvinyl chloride, and cellulose nitrate.

Using the bubble measurements for predicting compatibility, as illustrated in Figs. 2, 3, 4, and Tables III and V, the solids such as glass (0 deg.) and Buna-N rubber (12 to 15 deg.) are polar and should be compatible. Buna-S is only slightly polar (27 to 30 deg.) and should not adhere as well to the polar glass surface. Also, Durez phenolic resin (12987), although it has a 20 to 21 deg.

angle of contact, should not be as suitable for adherence of Buna-S (62 to 62 deg.) to glass (0 deg.) because of the wide difference in contact angle. The Durez 12687 resin with a 48 to 50-deg. angle of contact would be more suitable for adhering the moderately nonpolar Buna-S surface. Adhesion tests show that in one case the Durez 12987 failed to adhere to the Buna-S rubber, but it was held by the glass. In the other fase Durez 12687 failed at the glass surface and was held very tenaciously by the Buna-S surface.

To obtain adhesion, other materials such as a Hycar OR-25-1032X1, Polyblend 550X503, or Parlon are used to obtain the right degree of polarity by mixing with the Durez phenolic resins. By this blending of two resinous substances either by milling them together or by putting them into a common solvent it is possible to have a material of mixed polarity suitable for bonding the relatively nonpolar rubber surfaces to the polar-glass surface. In coating the mixed adhesive materials on the respective surface, a selective orientation of the adhesive molecules occurs with the polar portion being attracted to the polar surface and leaving the opposite polarity exposed. Figures 2(b), 2(c), and 2(d) indicate orientation has occurred by the difference in contact angles of the adhesive coated Buna-N, natural, and Buna-S rubber surfaces.

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Evaluation of Empirical Viscosity Measurements for Varnishes and Resin Solutions

By Maynard R. Euverard[1]

A Report of the Activities of Group 18, Subcommittee IX, of A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer and Related Products

THE entire subject of rheology, which is the science of flow properties of materials, is indeed a difficult one. Considering only the viscosity measurement problem of Newtonian or near-Newtonian materials, the work is not too difficult. Unfortunately, in the paint industry flow is governed not alone by viscosity but also by rheological properties which have been described as yield value, plastic flow, pseudo plastic flow, dilatant flow, tack, etc. It was concluded in the early days of the activities of Group 18, Subcommittee IX, that it would be practically impossible for such a group to consider rigorously the entire subject, taking into consideration all of these factors. There were two prime reasons for this decision: First, suitable instrumentation to measure all of these different properties are not at present widely enough dispersed in the industry to permit adequate cooperative work, and even if it were, this type of activity borders on long-range research which this group is not capable of sponsoring. Second, there is insufficient background and experience to establish A.S.T.M. methods for the purpose of interpreting and applying to production procedures, the information that might be gained by measuring all of these different attributes of rheology. Therefore, for future activity it was decided to limit the group's work to Newtonian or near-Newtonian materials.

During the course of the activities of Group 18, a wealth of information has been collected on many different types of measuring devices. Recent surveys have shown that although all of these devices are used in the industry, only a relatively few are used in sufficient number to justify establishment of A.S.T.M. methods. Even so, after presenting the surveys to the subcommittee for review and comment, it was requested that the accumulated information be printed so that it would be

available for all of those who have an interest in this subject. Accordingly, the purpose of this paper is to present the information that was on hand prior to the formation of Group 18, as well as that collected by the group, so that there will be available in one place a reference to the instrumentation and methods that are used in the industry for the determination of this all-important dimension of measurement. With this information so presented, Group 18 will complete its work on the revision of the present Gardner-Holdt tube method and will in the near future make recommendations as to other methods of viscosity measurement that should be included in the A.S.T.M. literature.
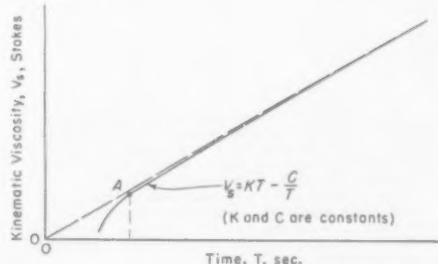
Fig. 1.—Typical Time-Viscosity Curve for Efflux Type Viscosity Cup.

GARDNER-HOLDT VISCOSITY TUBES

Gardner-Holdt viscosity tubes are widely employed at the present time. Recently there has been considerable discussion and disagreement on whether or not these tubes indicate absolute viscosity in poises or kinematic viscosity in stokes. So long as the density of the material that is being measured in these tubes is in the neighborhood of 1.0, the two are practically the same, but when this is not the case errors of considerable magnitude will accrue, since the stoke is defined as the ratio of poise viscosity to density. It is now the consensus of Group 18 that the Gardner-Holdt tubes indicate kinematic viscosity in stokes instead of absolute viscosity in poises, as has been accepted in the past. In line with this conclusion, Group 18 has submitted the necessary changes to be incorporated in A.S.T.M. Methods of Testing Varnishes (D 154 – 47).[2]

[2] 1949 Book of A.S.T.M. Standards, Part 4, p. 263.

EFFLUX TYPE VISCOSITY CUP

The laboratories of the Interchemical Corp. have over a period of years collected a considerable amount of data on different models of efflux type viscosity cups. This information was made available in a paper (unpublished) which was presented before the Lacquer Committee of the National Paint, Varnish and Lacquer Assn., April 9, 1948, at Roanoke, Va. In this paper it was shown that the efflux type viscosity cup measures kinematic viscosity.

In order for any apparatus to be considered more than just a gadget for making comparative measurement, it must furnish results which are in some way related to fundamental units. Figure 1 shows the relationship of time in seconds, of a material being measured in a representative cup, to kinematic viscosity in stokes.

The mathematical formula which fits this curve is also shown. It will be noted from the curve that above a given minimum time value (A) the curve is practically linear. This linearity falls off at the lower portion of the curve due to turbulence that is developed at the orifice when determinations are made on
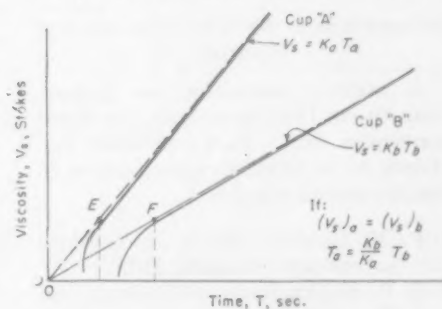
Fig. 2.—Representative Time-Viscosity Curves for Two Representative Cups.

materials of low viscosity. It will also be noted from the formula that if the time values are high the second term of the formula is of no consequence since the extrapolated straight-line portion of the curve would pass through the origin and be represented by the equation $V_s = KT$. If a minimum time for each type of viscosity cup is established, so that all determinations are conducted on the straight-line portion of the curve, a single multiplier can be

assigned for the time in seconds obtained in any particular cup which will convert that time to kinematic viscosity in stokes. About 40 different types of efflux viscosity cups have been studied, and their constants have been determined, so that it is possible to convert time in seconds in any particular cup to kinematic viscosity in stokes.

This conversion has also been carried one step further so that it is possible to convert from time in seconds in any one of these cups to time in seconds in any other cup.

Figure 2 shows typical time-viscosity curves for two representative cups. Again the linear portion of the curves above a given minimum (E and F) will be noted. Consider only the linear portion of the curves. Now if a correct conversion factor for cup A is chosen, its curve can be superimposed on that for cup B. Now what does this mean mathematically speaking? Figure 2 shows the mathematical equations for the curves obtained using each cup, in which the last terms have been omitted. Again this is permissible if only the straight-line portion of the curves is considered. If the same material is measured in each of these cups, of course the viscosity in each case is the same, and we can equate the right-hand terms of the equations also and solve the new equation for time in seconds in cup A. It is found to be equivalent to time in seconds in cup B times the constant of cup B, divided by the constant of cup A.

Table I shows the minimum time values and the constants for each of the 40 cups. This table also refers to the mode of operation of each type of cup.

INVESTIGATION OF CUP CONVERSION FACTORS

A working committee was formed from Group 18 to investigate the above conversion data. This committee was chosen to be broadly representative of the industry as possible:

P. N. Gardner, Henry A. Gardner Laboratory, Bethesda, Md.

E. B. Slaght, Resinous Products and Chemical Corp., Philadelphia, Pa.

M. R. Lipkin, Sun Oil Co., Norwood, Pa.

W. S. Hodgkiss, Sun Chemical Corp., Long Island City, N. Y.

R. L. Terrill, Spencer Kellogg and Sons, Inc., Buffalo, N. Y.

R. W. Charles, Congoleum-Nairn, Inc., Kearny, N. J.

N. T. Phelps, Sinclair Refining Co., Marcus Hook, Pa.

Each of the above members was furnished samples of two different oils of known viscosity and was requested to

TABLE I.—VISCOSITY CUP CORRELATION DATA.

Cup	Factor K^a	Minimum Time	Method of Reading
American Can	3.5	35	e
A.S.T.M. 0.07	1.4	60	b
A.S.T.M. 0.10	4.8	25	b
A.S.T.M. 0.15	21	9	b
A.S.T.M. 0.20	61	5	b
A.S.T.M. 0.25	140	4	b
A. & W. "B"	18.5	10	c
A. & W. Crucible	11.7	12	d
Caspers Tin Plate	3.6	39	e
Continental Can	3.3	12	b
Crown Cork and Seal	3.3	12	b
Engler	7.3	18	...
Ford No. 3	2.4	34	e
Ford No. 4	3.7	23	e
Murphy Varnish	3.1	24	f
Parlin 7	1.3	60	b
Parlin 10	4.8	21	b
Parlin 15	21.5	10	b
Parlin 20	60	5	b
Parlin 25	140	15	b
Parlin 30	260	10	b
Pratt Lambert A	0.61	70	b
Pratt Lambert B	1.22	60	b
Pratt Lambert C	2.43	40	b
Pratt Lambert D	4.87	25	b
Pratt Lambert E	9.75	15	b
Pratt Lambert F	19.5	9	b
Pratt Lambert G	38	7	b
Pratt Lambert H	76	5	b
Pratt Lambert I	152	4	b
Redwood	0.23	320	...
Saybolt Furol	2.1	17	g
Saybolt Universal	0.21	70	g
Scott	1.6	20	h
Westinghouse	3.4	30	f
Zahn G-1	0.75	50	d
Zahn G-2	3.1	30	d
Zahn G-3	9.8	25	d
Zahn G-4	12.5	14	d
Zahn G-5	23.6	12	d

a The conversion factors given provide approximate values in centistokes when the cup reading is above the minimum time in seconds indicated.
b Cup is filled level full and time is determined for the first 50 ml. to flow through the orifice.
c Cup is filled level full and time is measured for complete emptying, determined by observing from the inside of the cup the entrance into the orifice of the hydrostatic head of the material being measured.
d The cup is submerged in the material to be measured and is permitted to become completely filled. The time of efflux is determined from the instant of quickly raising the cup out of the viscous material to the break in the stream at the orifice.
e The cup is filled level full and efflux time is determined from the start of flow to the break of the stream at the orifice.
f Cup is filled to mark on inside and time is determined for complete emptying, as noted by break in stream at the orifice.
g Cup is filled level full and time is determined for the first 60 ml. to flow from the cup.
h 200 ml. of material is placed in the cup and the time for the first 50 ml. to flow from the cup is determined.

make a measurement on each sample with different types of available viscometers.

Table II gives a correlation of the data that were submitted by the working committee. The following conclusions were made on the basis of this work.

1. All methods reported on afford a measure of viscosity within acceptable limits of accuracy so far as any one operator is concerned, and so long as comparative results only are considered.

2. There is insufficient inherent accuracy built into many of the devices. This makes accurate exchange of information impossible even when different

devices of the same type are employed. There are known factors influencing some of these values. This is especially true of the Ford No. 4 cup and the large deviation that is indicated should not mitigate acceptance by the industry of a "standard" Ford type cup.

During the time that the working committee of Group 18 was collecting its data the laboratories of Interchemical Corp. determined both constants for the cup formula that is shown in Fig. 1 so that it is possible to convert seconds, in any cup that has been investigated, to kinematic viscosity in stokes without having to consider the "Minimum Time" shown in Table I. All of these constants are given in Table III.

It is concluded from the data submitted by members of the working committee that the conversion factors that were determined for the different types of viscosity cups were of sufficient accuracy for most measurements, which are satisfied by the efflux type viscosity cups, or other instruments which are of a production control nature. It is true that rather wide discrepancies are indicated for several of the devices, but in most cases the reasons for these discrepancies are known, and do not reflect adversely on the conversion constants.

Care must be exercised in using the above conversion information. Relationships that have been developed are based on results obtained on mineral oils which displayed negligible non-Newtonian properties. Errors will be unavoidable on materials having other characteristics when conversion is attempted between widely dissimilar cups.

FORD CUP STANDARDIZATION

A project has been in progress for some time in the Standards and Methods of Test Committee of the Chicago Club of the Federation of Paint and Varnish Production Clubs. R. R. Bruhn of the Sherwin-Williams Co., who was responsible for this work in the Chicago Club, presented the complete findings on this project at the 1949 Convention of the Federation of Paint and Varnish Production Clubs.[3]

INTERCHEMICAL INCLINED TUBE VISCOMETER

A paper dealing with an inclined tube viscometer was presented by the author[4] at the 1947 Spring Meeting of the Society in Philadelphia. The purpose of

[3] R. R. Bruhn, "The Standardization of the Ford Type Cup," Official Digest of the Federation of Paint and Varnish Production Clubs, No. 298, November, 1949, pp. 831–837.
[4] M. R. Euverard, "Interchemical Inclined Tube Viscometer," Symposium on Paint and Paint Materials. Philadelphia Spring Meeting. Am. Soc. Testing Mats., p. 85 (1947). (Symposium issued as separate publication STP No. 15.)

TABLE II.—COMPARISON OF VISCOSITY MEASURING DEVICES.

Device	Oil	Number of Determinations	Average Value	Number of Instruments	Per cent Spread[c]	Average Deviation from Average[c,e]	Number of Observers	Average Individual per cent Spread[c,f]	Average Value Converted to Stokes	Source of Conversion Information[g]	Per cent Error
A. & W. "B"	A[a]	3	17.0	1	..	..	1	0	3.05	(1)	+5
	B[b]	3	44.0	1	..	..	1	1	8.41	(1)	0
A.S.T.M. 0.07	A	6	241.1	2	7	1	1	4	3.06	(1)	+5
	B	2	666.5	2	10	5	1	..	8.48	(1)	+1
A.S.T.M. 0.10	A	3	67.1	1	..	..	1	0	3.12	(1)	+7
	B	3	192.6	1	..	..	1	3	9.56	(1)	+14
A.S.T.M. 0.15	A	6	15.7	2	15	6	1	3	3.00	(1)	+3
	B	6	41.0	2	14	5	1	3	8.75	(1)	+4
A.S.T.M. 0.20	A	6	6.1	2	10	2	1	5	3.40	(1)	+17
	B	6	16.1	2	7	3	1	2	10.3	(1)	+22
A.S.T.M. 0.25	A	6	3.2	2	6	0	1	3	3.34	(1)	+15
	B	6	7.0	2	13	4	2	5	9.85	(1)	+18
Demmler 1	A	4	84.0	1	..	..	2	1	2.68	(2)	−8
	B	4	245.4	1	..	..	2	3	7.82	(2)	−13
Demmler 10	A	4	9.7	1	..	..	2	6	3.30	(2)	+13
	B	4	26.0	1	..	..	2	2	8.50	(2)	+1
Durez	A	4	223.1	1	..	..	2	0	...	(3)	..
	B	..	....	..	..	..	..	..	...	(3)	..
Ford No. 3	A	10	124.3	3	24	9	4	1	3.12	(4)[h]	+7
	B	10	328.0	3	22	8	4	3	8.10	(4)[h]	−4
Ford No. 4	A	20	58.3	7	71	22	8	1	2.2	(1)	−24
	B	20	161.1	7	87	25	8	1	6.1	(1)	−27
Saybolt Furol	A	3	138.3	1	..	..	1	1	2.93	(6)	+1
	B	0	....	..	..	..	..	..	...	(6)	..
Sinclair	A	3	54.4	1	..	..	1	3	...	(3)	..
	B	3	156.2	1	..	..	1	10	...	(3)	..
Zahn G-1	A	3	197.0	1	..	..	1	1	1.80	(5)[h]	−38
	B	0	....	..	..	..	..	..	...	(5)	..
Zahn G-2	A	7	98.9	2	25	11	3	2	2.91	(5)[h]	0
	B	3	311.6	1	..	..	1	4	9.85	(5)[h]	+17
Zahn G-3	A	6	31.6	1	..	..	2	1	2.80	(5)	−4
	B	6	88.0	1	..	..	2	1	9.28	(5)	+10
Zahn G-4	A	4	21.5	1	..	..	2	1	2.50	(5)	−14
	B	4	56.9	1	..	..	2	1	7.70	(5)	−8
Gardner-Holdt Tubes	A	16	K+	6	9	..	6	..	2.80	(7)	−4
	B	16	V−	6	14	..	6	..	8.36	(7)	−1
Gardner Visco-meter	A	3	3.10	1	..	..	1	3	3.10	(7)	+7
	B	3	8.20	1	..	..	1	5	8.20	(7)	−2
Interchemical Inclined Tube	A	22	2.69	8	5	1	5	1	2.69	(7)	−..
	B	19	7.82	8	13	3	5	0	7.82	(7)	−7
Hoeppler	A	3	2.61	1	..	..	1	0	2.79	(8)	−4
	B	3	7.55	1	..	..	1	0	8.11	(8)	−4
Brookfield	A	18	2.60	4	17	3	5	0	2.78	(8)	−4
	B	19	7.36	4	9	2	5	1	7.90	(8)	−6
Interchemical Rotational	A	1	2.75	1	..	..	1	..	2.94	(8)	+1
	B	1	8.30	1	..	..	1	..	8.91	(8)	+6
Interchemical Tackmeter	A	1	3.50	1	..	..	1	..	3.74	(8)	+29
	B	1	9.00	1	..	..	1	..	9.67	(8)	+15
Stormer, Bob (sec. per gr.)	A	3	26.0/350	1	..	..	1	2	2.85	(4)	−2
	B	3	28.9/850	1	..	..	1	3	7.68	(4)	−9
Stormer (Krebs units)	A	3	57.1	1	..	..	1	1	...	(9)	..
	B	3	81.1	1	..	..	1	1	...	(9)	..
Stormer, Stroboscopic	A	3	89.2	1	..	..	1	1	...	(3)	..
	B	3	196	1	..	..	1	1	...	(3)	..
Sun Rotational	A	3	2.61	1	..	..	1	2	2.79	(8)	−4
	B	3	7.27	1	..	..	1	1	7.81	(8)	−7
Capillary Tubes	A	2	2.88	1	..	..	1	..	3.08	(8)	+6
	B	2	8.19	1	..	..	1	..	8.80	(8)	+5

[a] Viscosity, 2.91 stokes; density, 0.936 g. per ml.
[b] Viscosity, 8.41 stokes; density, 0.931 g. per ml.
[c] To better understand how tabulated values were evaluated:
Let A = highest reading reported by any cooperator.
B = lowest reading reported by any cooperator.
C = average reading reported by all cooperators.
a_x = highest reading reported by an individual x.
b_x = lowest reading reported by an individual x.
c_x = average reading reported by an individual x.
[d] Value is $(A − B)/C$.
[e] Values is average $(C − c_x)/C$ without regard to sign.
[f] Value is $\frac{1}{n} [(a_1 − b_1)/c_1 + (a_2 − b_2)/c_2 \ldots + (a_n − b_n)/c_n]$
[g] Source:

(1) Interchemical Corp. calibration data.
(2) *Organic Finishing*, Vol. 6, No. 10, October, 1945.
(3) No information available.
(4) Gardner, "Physical and Chemical Examination of Paints, Varnishes, Lacquers."
(5) *Organic Finishing*, Vol. 6, No. 6, June, 1945.
(6) A.S.T.M. Method D 666-44, 1949 Book of A.S.T.M. Standards, Part 5, p. 972.
(7) Manufacturer's literature.
(8) Stoke = poise/density.
(9) No director conversion possible.
[h] Extrapolated.

the paper was to describe a new, more rapid, and accurate means of viscosity measurement than was practicable with other equipment.

The advantages listed for the Interchemical inclined tube viscometer were as follows:

1. The required equipment is simple and sturdy.
2. Results are obtained on a continuous scale in fundamental units.
3. Only one standard tube is required for use with the instrument, and it is employed for calibration purposes only.
4. Erroneous measurements due to temperature variation outside the control range of the constant temperature bath are eliminated. Viscosity can be determined at any temperature within the range of the liquid bath.

5. The equipment is very accurate. Operator error can be practically eliminated by averaging multiple readings.
6. Wide measuring ranges are inherent in the apparatus.
7. The cost of the inclined tube viscometer is nominal.
8. The time required for viscosity measurement is short compared to most other measuring devices.
9. Only a small amount of any test specimen is required.
10. The test specimen is enclosed in a tube during measurement obviating errors due to evaporation.

Subsequent to the development of the Interchemical inclined tube viscometer, it was pointed out by Euverard and Hurley,[5] that surface tension is a factor

[5] M. R. Euverard and Hurley, D. R., "Surface Tension Measurement," *Analytical Chemistry*, Vol. 21, p. 1177 (1949).

in the measurement of viscosity in any air bubble type instrument. Fortunately, experience indicates that for most purposes this factor can be neglected, either because most of the materials measured are in the same surface tension range or because greater importance in our industry is placed on comparative rather than absolute measurements.

The fact that the inclined tube viscometer has not received very wide acceptance in the industry to the present time is easily understood since there is such a great variety of instruments available. It is confusing for anyone, who wishes to buy a piece of equipment for any particular purpose, not to find readily in the literature definite recommendations as

TABLE III.—EVALUATION OF VISCOSITY CUP FORMULA CONSTANTS K AND C.

Cup	K	C	Source[b]
American Can...	0.03727	12.	(1)
A.S.T.M 0.07...	0.01273	6.0	(1)
A.S.T.M. 0.10...	0.04966	8.0	(1)
A.S.T.M. 0.15...	0.21429	5.0	...
A.S.T.M. 0.20...	0.64706	3.0	(1)
A.S.T.M. 0.25...	1.4615	3.3	(1)
A. & W. "B"....	0.1907	3.5	(1)
A. & W Crucible	0.1222	4.0	(1)
Caspers Tin Plate	0.03744	15	(1)
Continental Can.	0.0361	1.0	(1)
Crown Cork and Seal.........	0.0361	1.0	(1)
Engler[a].........	0.00147	3.74	(2)
Ford No. 3......	0.024667	8.0	(5)
Ford No. 4......	0.0378	4.0	(1)
Murphy Varnish.	0.03056	3.0	(1)
Parlin No. 7.....	0.01273	6.0	...
Parlin No. 10....	0.04966	8.0	...
Parlin No. 15....	0.21429	5.0	(4)
Parlin No. 20....	0.64706	3.0	...
Parlin No. 25....	1.4615	3.3	...
Parlin No. 30....	2.727	60	(4)
Pratt&Lambert A	0.00618	96	(5)
Pratt&Lambert B	0.01236	48	(5)
Pratt&Lambert C	0.02472	24	(5)
Pratt&Lambert D	0.04943	12	(5)
Pratt&Lambert E	0.09886	6.0	(5)
Pratt&Lambert F	0.19772	3.0	(5)
Pratt&Lambert G	0.39544	1.5	(5)
Pratt&LambertH	0.79088	0.75	(5)
Pratt&Lambert I	1.5818	0.38	(5)
Redwood.......	0.0026	1.88	(2)
Saybolt Furol...	0.02125	1.5	(6)
Saybolt Universal	0.00217	1.5	(7)
Scott.........	0.01639	2.0	(1)
Westinghouse....	0.03520	8.0	(5)
Zahn G-1.......	0.0080	4.0	(3)
Zahn G-2.......	0.02929	5.0	(3)
Zahn G-3.......	0.10667	18.0	(3)
Zahn G-4.......	0.1400	12.0	(3)
Zahn G-5.......	0.2325	2.0	(3)

[a] Valve of t used is degrees Engler $\times$ 5.13.
[b] Source:
(1) Interchemical Corp. Experimental data.
(2) "Blackmer Rotary Pumps," Engineering Manual, November 3, 1944.
(3) *Organic Finishing*, Vol. 6, No. 6, June, 1945, pp.
(4) *Organic Finishing*, Vol. 6, No. 9, September, 1945, pp.
(5) Gardner, "Physical and Chemical Examination of Paints, Varnishes, Lacquers."
(6) A.S.T.M. Method D 666-44, 1949 Book of A.S.T.M. Standards, Part 5, p. 972.
(7) A.S.T.M. D 446-39, *Ibid.*, p. 110.

to the best type of instrumentation. It is believed that this new device is an improvement and will receive closer attention when a better and more thorough understanding is developed on the subject of viscosity measurement and viscosity control.

SURVEY OF VISCOSITY MEASUREMENT METHODS USED BY MEMBERS OF SUB-COMMITTEE IX

In order to define better the activities of Group 18 it was considered advisable to circulate a questionnaire to the membership of Subcommittee IX on Varnishes to determine more adequately the need and the preference of the industry for viscosity measuring equipment for varnishes and resin solutions. This survey was conducted by J. C.

TABLE IV.—SUMMARY OF QUESTIONNAIRE ON A.S.T.M. POLICY OF EFFLUX VISCOSITY CUPS.

Number of Questionnaires Issued.....62
Number of Replies Received........34 (These were mostly from paint and varnish manufacturers.)
Number Unreturned.................28 (These had been sent to mostly institutional and raw material representatives.)

1. 28 "Yes" and 3 "No" votes approve in principle the adoption of a test method for an efflux cup, applicable primarily to black and other nontransparent varnishes, and also applicable, if possible, to pigmented coating materials.

2. Three choices of efflux cups are as follows:

First Choice	Second Choice	Third Choice	
24	5	..	Ford No. 3 and Ford No. 4
3	9	6	A.S.T.M. D 333 (N/C lacquers)
2	3	4	Saybolt Furol and Universal
0	2	6	Zahn
0	0	1	Ault and Wiborg
0	1	0	Stormer
2	3	5	*New* A.S.T.M. design

3. In the event his first choice being adopted by A.S.T.M., the individual replying noted whether he would promote its use in his organization in:

YES	NO		
20	6	(a)	Nontransparent varnishes for routine control.
16	4	(b)	Nontransparent varnishes for umpire testing.
19	6	(c)	Pigmented coatings for routine control.
15	3	(d)	Pigmented coatings for umpire testing.

4. Organizations are now using as preferred viscosity testing devices:

Viscosity Testing Device	Transparent Varnish		Non-transparent Varnish		Enamels Non-thixotropic		Paints Thixotropic	
	Routine	Umpire	Routine	Umpire	Routine	Umpire	Routine	Umpire
Gardner-Holdt tubes	26	18						
Interchemical viscometer								1
Interchemical inclined tube	1	1						
A. & W. tubes and cup	1		1		1			
Pratt & Lambert tubes and cup	1		1		1			
Ford No. 3 and No. 4 cups	3	2	18	17	13	13		1
Saybolt universal and furol		1	1	1				
A.S.T.M. D 333 lacquer cups			1		1	1		
Parlin			1	1	1	1		
Zahn			1	1	1	1		
Sherwin-Williams (5 orifices)			1		1			
Brookfield		4	4	6		2	4	3
Stormer	1	1	4	4	18	12	23	18
MacMichael (G.E. modification)		1	1	1	1	1	1	1
Gardner mobilometer			1	1	1	1	2	2
Hoeppler viscometer		1			1			
Brabender								1

Weaver and a correlation of his findings is given in Table IV.

It will be observed from this survey that the Gardner-Holdt viscosity tubes and the Ford type cups are preferred by the majority, the Gardner-Holdt tubes being used for transparent varnishes and the Ford cups being used for nontransparent varnishes and enamels.

CONCLUSIONS

Based upon the information in this paper it is believed that Group 18 has two remaining tasks: (1) To complete the present work on the Gardner-Holdt tubes, and to revise as may be considered necessary the present A.S.T.M. method D 154 – 47. (2) To draft a test method for the Ford type cup for inclusion in A.S.T.M. literature.

Caution should be exercised in employing the conversion data that have been presented. Unconsidered use of these data may lead to erroneous results on different products or formulations which display characteristics deviating widely from the Newtonian type materials employed in this work.

I wish to take this opportunity to acknowledge the interest and the efforts of all members of Group 18 and all others who have assisted in this work. Especially do I want to thank those who have served on the Working Committees.